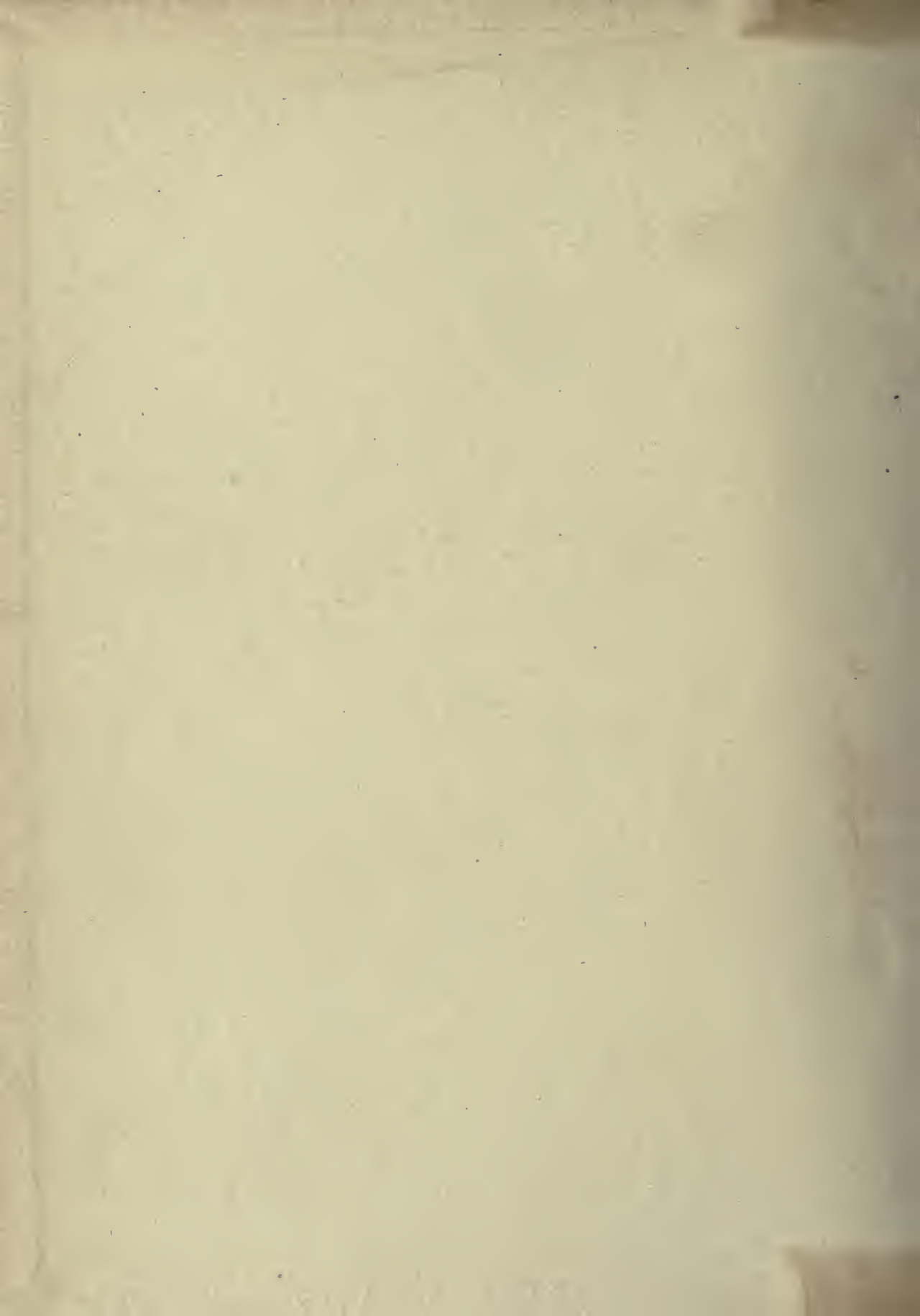






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THE CHEMICAL NEWS, JULY 9, 1897.

THE
CHEMICAL NEWS

AND
JOURNAL OF PHYSICAL SCIENCE.

WITH WHICH IS INCORPORATED THE "CHEMICAL GAZETTE."

A Journal of Practical Chemistry

IN ALL ITS APPLICATIONS TO

PHARMACY, ARTS, AND MANUFACTURES.

EDITED BY
WILLIAM CROOKES, F.R.S., &c.

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THE CHEMICAL NEWS.

VOLUME LXXV.

EDITED BY WILLIAM CROOKES, F.R.S., &c.

No. 1936.—JANUARY 1, 1897.

SEA-WATER MICROBES IN HIGH LATITUDES.

By E. FRANKLAND, D.C.L., F.R.S.,
and W. T. BURGESS, F.I.C.

THE bacteriology of sea-water is beginning to receive deserved attention from biologists, for it lies at the very foundation of the marine flora and fauna, as bacteria doubtless form the initial food of higher marine organisms; unless indeed there be a still more minute world of living matter too small for discovery by our present modes of investigation.

In the year 1892, Mr. H. L. Russell examined at the biological station in Naples, a number of samples of sea-water collected at various depths in the Bay of Naples. The water at or near the surface was found to contain a number of colonies per c.c., varying from 64 at four kilometres from land, to six at fifteen kilometres. There was, however, no constant relation between the numbers of bacteria and the distance from land; thus, at eleven kilometres from land 78 per c.c. were found. On the other hand, Sanfelice and de Giaxa found a rapid reduction on receding from the shore, but only within a distance of three kilometres.

In 1894 M. Cassedebat studied the action of sea-water upon pathogenic organisms, and found that in sterilised sea-water *Staphylococcus aureus* died in twenty-two to twenty-four days, *Citrus* in nineteen to twenty-two days, *B. Friedländer* in thirty-five to forty days, *B. Anthracis* in twenty-one to twenty-four days, *B. of green diarrhæa* in sixteen to twenty days, *Spirillum Deneke* in twenty-two to twenty-five days, *Proteus vulgaris* in twenty-three to twenty-six days. The cholera bacillus lived more than thirty-five days, but the typhoid bacillus died in forty-eight hours.

The most complete bacteriological investigation of sea-water hitherto undertaken was made by Drs. Bernhard Fischer and E. Bassenge, and the results are published in the *Centralblatt für Bacteriologie*, 1894, xv., 657. They include microbe determinations in the waters of the Atlantic, English Channel, the Baltic, and the North Sea. The places where the samples were taken range from 8° S. Lat. to 60° N., and were collected at various seasons during the celebrated Plankton expedition, and on a voyage to and from the West Indies. The plate cultures were made with gelatin containing 2 per cent of agar-agar dissolved in a little sterilised sea-water, and the final

investigation of the pure cultures was made in the Bacteriological Institute at Kiel.

The maximum number of microbes found at the surface was 29,400, the minimum 0, and the mean in 175 samples 1083. The mean was only surpassed 26 times; seven samples developed no organisms, fifty-seven developed from one to twenty-five colonies, seventeen from twenty-six to fifty, and fourteen from fifty-one to one hundred colonies per c.c. The highest numbers were found near land, thus confirming, in this respect, the observations of Sanfelice and de Giaxa in the Bay of Naples. It was also found that sunshine affected the number of bacteria at the surface.

During the recent solar eclipse expedition to Vadsö, we were able to extend these observations up to 71° N. lat., Captain Eilertsen, of the Bergen Company's steamer *Neptun* kindly stopping the ship whilst we took the samples. The water for bacteriological examination was collected at about 2 feet from the surface in glass tubes (previously exhausted, sealed, and sterilised) by means of the apparatus devised by one of us and described in the *CHEM. NEWS*, lxx., p. 54. The colonies were counted after about five days incubation at 20° C.

Sample No. 1: Vest Fjord, five miles from land, lat. 68° N., at 6 a.m. Aug. 5, 1896; No. of colonies, mean of two cultivations, 51 per c.c.

Sample No. 2: Off Løppen, about three miles from land, lat. 70° 12' N., at 10 a.m. August 6, 1896. Unfortunately, the note of the number of colonies counted, in duplicate cultivations of this and of the following sample, has been lost; but the average number in each case did not differ materially from that obtained from the Vest Fjord water.

Sample No. 3: Off the North Cape, about two miles from land, lat. 71° 10' N., temperature of sea 7·8° C., 1 a.m. August 7, 1896. A blank culture plate, similarly treated in every way, did not yield a single colony.

No sample was taken in the Väranger Fjord on account of proximity to land and the towns of Vardö and Vadsö. The temperature of the sea in the Fjord at 7 a.m. on August 8 off Vadsö was 9·0° C.

We have to apologise for this very fragmentary record of bacterial life in the Arctic Ocean, but we trust that it may have the effect of directing the attention of arctic and antarctic voyagers to the subject; for, in view of the important relations existing between microbes and animal life in the arctic regions, it is very desirable that these

observations should be carried on up to higher latitudes, especially where the temperature of the sea does not rise above 0°C .

ON THE SPECTRA OF COPPER, SILVER, AND GOLD.*

By J. M. EDER and E. VALENTA.

OUR former preliminary research had shown that the elements copper, silver, and gold give continually new results on the variable spectra of the elements, for these elements yield spark-spectra extremely rich in lines for surpassing the corresponding arc-spectra in number and sharpness. These researches were commenced two years ago, but could not then be completed, as the spectrograph with a quartz prism at the disposal of the authors had too slight a dispersion in the less refrangible regions, and the spectrograph with a compound glass prism supplied this deficiency in the blue and the violet as far as the beginning of the ultra-violet, but was not sufficiently effective in the visible part.

We obtained two excellent Rowland's concave gratings with a very short focus, giving an admirable definition if the light was very strong. We selected one which produced the spectra of the first, second, and third order with great clearness. The spectra on both sides of the grating never show a perfectly equal brightness.

The ultra-violet of the first order, of $\lambda = 3900$, at about 2500 is moderately bright, but from $\lambda 2500$ very faint. The spectrum of the second order is decidedly darker on the red, the yellow, and the green than the spectrum of the first order, which is in this region three times brighter than the latter. On the contrary, the brightness of the spectrum of the second order is very great. From $\lambda 2800$ to $\lambda 1900$ we worked with the quartz spectrograph with one prism, which in these regions is far superior to the grating spectrograph in brightness and in resolving power. The spectrum of the third order is with our grating very bright; in the ultra-violet of $\lambda 2200$ it is about equal in brightness to the ultra-violet of the second order, perhaps somewhat brighter. Consequently the spectrum of the second order is intersected by very luminous lines of the third order, which require to be separated out and identified, and then furnish an exceedingly sharp control for the measurements of the spectrum of the second order. The violet and ultra-violet near the Fraunhofer line H come up so strongly in the spectrum of the third order as even to penetrate light yellow glass, and can be eliminated only by dark yellow glasses or concentrated strata of picric acid. The spectrum of the fourth order was plainly visible in the grating which we used, but its brightness is small.

The authors reproduce an important illustration from Ames's memoir, "The Concave Grating in Theory and Practice," taken from the *Johns Hopkins University Circular*, No. 273, 1889.

As a source of error in working with the grating-spectrograph are mentioned the so-called ghostly lines, which sometimes appear as rather sharp lines at almost equal distances, or sometimes as a unilateral expansion of very intense lines.

1. The copper lines 5782, 5218, and 5105 correspond to the lowest temperature prevailing in the Bunsen flame.
2. The same lines with an accession of Cu $\lambda = 5700$, 5292, 5153, 4704, 4651, and 4275, pertain to the rather higher temperature of the faint sparks springing on between electrodes saturated with copper chloride.

3. All the lines occur in the arc-spectrum, and at the highest temperatures of the arc-spectrum, and must consequently be considered as constant lines peculiar to copper at the most variable temperatures.
4. In the arc-spectrum there occur far more lines than in the cases described under 1 and 2, which holds good especially for the more strongly refrangible lines. As especially characteristic lines, in addition to those mentioned under 1 and 2, we mention $\lambda = 4062, 3308, 3275, 3247, 2392$.
5. In the spark-spectrum all the main lines mentioned under 1, 2, and 3 occur also as principal lines. In addition there appear many very intense new lines which are wanting in the arc- and the flame-spectra, whilst some strong Cu-lines of the arc-spectra recede or disappear at the high temperature of the spark of the Leyden jar.

As regards these phenomena, the Cu-spectrum must be regarded as very variable according to the temperature.

Silver, even in the strongest Leyden jar spark, is less luminous than copper or gold. When the Ruhmkorff spark strikes over, the atmospheric lines come up very strongly, so that we were compelled to work in an atmosphere of hydrogen. But even in this case the subordinate rays of the Ag-spectrum were but feebly luminous. A number of the silver lines in the green part of the spectrum are extended into bands, but there are many sharp lines, and in the more refrangible part of the spectrum sharp lines predominate.

The spectrum of silver must be considered as variable, since it becomes extremely rich in lines by the accession of intense silver lines in the hot Leyden spark. The moderately bright lines of the arc-spectrum are mostly retained, but are often surpassed in intensity and sharpness by the new lines of the spark-spectrum.

The spectrum of gold is less known than the spectra of copper and silver. The lines of Lecoq, $\lambda = 5601, 5230, 5210, 4437, 4338$, and 4064, do not belong to gold. 5601 and 5210 belong to palladium, 5228 and 4442 to platinum, and 4355 to air (nitrogen).

Krüss was in error in ascribing the line 4064 (more correctly 4065) to nitrogen, since it appears distinctly in an atmosphere of pure hydrogen.

The authors have observed 660 lines in the spark-spectrum of gold, 50 of which are common to the spark and arc spectra, but more than 500 are new lines.

CALCIUM CARBIDE: A NEW REDUCING AGENT.

By H. N. WARREN, Principal, Liverpool Research Laboratory.

SINCE the introduction of this remarkable substance, it is significant that scientific men have been content to allow the product to rank solely as a water decomposer, and thus regard the production of acetylene the only available product.

Researches of a somewhat lengthy description, which have lately been carried out at the above laboratory, involve the use of calcium carbide as a metallurgical reducing agent.

In the first instance an excess of litharge was heated to redness in contact with the carbide, in a clay crucible, the reaction being accompanied by vivid incandescence, resulting in the formation of metallic lead and calcium oxide, CaO.

A further portion was now selected, in which the proportion of carbide exceeded that of the litharge; this was further subdivided into various smaller portions, each portion being exposed to various temperatures, resulting in a regulus of calcium and lead of varying percentage, together with the expulsion of CO_2 .

* An especial reprint from the *Transactions of the Mathematical and Natural Science Class of the Imperial Academy of Sciences*, Vienna, 1896.

The alloys thus formed are all more or less brittle, and to a certain extent sonorous when struck, their melting-point ranking below that of pure lead, and are slowly, but completely, decomposed in contact with aqueous vapour, the reaction being much less energetic than that afforded by alloys of lead with the alkaline metals. Stannic oxide, cupric oxide, and also ferric oxide, at corresponding higher temperatures, were readily reduced, yielding results of no practical value; in the case of the cupric alloys those samples containing under 1 per cent of calcium being rendered cold-short and breaking under very small strain, whilst, on the other hand, iron containing calcium approaches in appearance that of ferro-manganese, being even more brittle, and very oxidisable in contact with water.

In a further operation, oxides of manganese, nickel, cobalt, and even chromium, molybdenum, and tungsten, were readily reduced, yielding calcium alloys. Results of experiments, comprising the reductive action of the carbide upon the earthy chlorides and their haloids, will be shortly to hand. The already partial success of these reactions seem to point most conclusively towards a new and powerful reducing agent, which at the same time, considering the market value of the carbide in question, could not fail to replace both sodium and potassium.

Liverpool Research Laboratory,
18, Albion Street, Everton, Liverpool.

THE MANUFACTURE OF CALCIUM CARBIDE.*

By J. T. MOREHEAD and G. de CHALMOT.

So universal is the interest in acetylene gas and so different the estimates and opinions as to the cost of calcium carbide as a source of the cheap production of acetylene gas, that we have thought it desirable to place on record the data from our actual experience in the production of calcium carbide in quantities. The works of the Willson Aluminium Company have been running night and day since May 1st, 1895, producing calcium carbide. These works are daily duplicating the results here given and can expand indefinitely. Each individual step, except water power, as taken at Spray, N. C., is capable of being changed in the direction of reducing the cost of the output, as these efforts have been attended with the clumsiness, lack of adaptability, and excessive cost that is incident to all efforts along an untrodden path. Still we can produce calcium carbide at less than 25 dols. per ton, including wear and tear and interest on capital.

Beyond looking after the dynamos, no special training is necessary, as neither metallurgical nor chemical skill is required in the operations. We grind and mix coke and lime, start the water wheel, see that the arc is formed, shovel in the mixture of lime and coke, and the volt- and ammeter show when to lower or raise the carbon pencils, which is done by means of a screw located in the dynamo room, away from the furnace. We can measure with an ordinary yard stick on this screw the height of the piece of carbide in the furnace. We stop when we have raised the carbon pencils 33 inches, switch the current off to another furnace and repeat the operation. The carbide in the former furnace, as soon as cooled and brought in contact with water, is all ready to do perfect work in generating acetylene gas; it will proceed with this work without help and will make room therefor in spite even of bands of steel.

Water power costs us 6 dols. per horse power. Water in the raceways ready for the water wheels is now offered in enormous quantities to the Willson Aluminium Company at the rate of 5 dols. per horse power per year.

* Read Sept. 3rd before the Springfield meeting of the A.A.A.S. by one of us (M.). We have made since then several additions, so as to make the article complete up to the present time. From the *Journal of the American Chemical Society*, April, 1896.

These powers are located at different places, where coke and lime can be had cheaply, and also cheap transportation for the carbide to the market.

The technical description of our process which follows herewith was written by G. de Chalmot, who has had for some time personal supervision of the operations of the Willson Aluminium Company.

In the year 1888 Mr. T. L. Willson started a series of experiments with a view of reducing refractory ores in the electric furnace, and among other valuable things he made calcium carbide.*

We will first give a short description of the furnace and a general outline of the process, then enlarge somewhat on the details. The furnace used in Spray, N. C., is built of ordinary brick (a sectional front view is given in Fig. 1). The front side is formed by four iron doors, the one above the other. The upper two remain closed usually. The chimney is attached near the top of the furnace, and commences with a flue, *m*, in the corner. The furnace measures at the bottom inside $2\frac{1}{2}$ by 3 feet. The electric current enters at the bottom and top. The bottom electrode is an iron plate, *a*, covered with 3 inches of carbon, *b*. For this covering we use pieces of carbon pencils or a mixture of coke and coal tar. Sixteen copper cables of 0.75 inch in diameter, *c*, convey the electricity from the dynamos to the bottom electrode.

Sixteen other cables are connected with the top electrode, *d*. The top electrode is composed of six carbon pencils, *e*, each 4 inches square and 36 inches long. Six pencils are arranged in three pairs behind each other, and are cut out at the top so as to fit in the carbon holder, *f*. They are enveloped together by a sheet of iron, *g*, which is shown in the right-handed furnace of Fig. 1. They really form one pencil. The carbon holder is screwed to a copper bar, *h*, which is three inches square, and to which the copper cables are connected. This bar is fastened by a chain that runs over two pulleys to a long upright screw, *i*. On this screw is a nut which forms the centre of a wheel, *k*. By turning the wheel the screw can be raised or lowered. The man who attends to the wheel has the volt- and ammeter before him. The electric current is generated in two dynamos to which transformers are connected, and which can give a current of from 50 to 100 volts. The power is furnished by a water wheel of 300 horse power under 28 feet fall.

Two of the furnaces have been working for twelve months, and they have given satisfaction, except for working not sufficiently economically. In the furnaces built for the Niagara Falls Carbide plant many changes which we suggested have been adopted, looking to economy of production. We give here a short description of these furnaces (Figs. 2 and 3).

In Spray it is necessary to allow the furnace to cool before emptying it. In order to use one and the same furnace continuously, the bottom of the furnace is replaced by an iron car, *a*, which runs on a track, and in which carbide is formed. When the car is filled the pencils, *b*, have been lifted entirely out of it. The current is then shut off, door *c* is opened, the full car is run out and replaced by an empty car. The pencils are lowered again to the bottom of the car and a new run is commenced.

The bottom of the car is covered with from 4 to 8 inches of carbon. When the contents of the car have sufficiently cooled outside the furnace, which will take from six to twelve hours, the body of the car is lifted from the track by the trunnions, *d*, and turned over. The con-

* We will note here that Moissan, who discovered this process for making carbide, independently of Mr. Willson, communicated incidentally, at the meeting of the French Academy of December 12th, 1892 (*Compt. Rend.*, 115, 1033) that a carbide of calcium is formed if calcium oxide is heated in an electric furnace with carbon electrodes. He investigated the compound much later (*Compt. Rend.*, 118, 50). Mr. Willson, who sent, during the summer of 1892, samples of carbide, for examination, to Lord Kelvin, of the Glasgow University, clearly antedates Moissan. See *Journal of Franklin Institute* of 1895, page 333.—Note.

tents are dropped on a grate formed of iron bars, on which the piece of carbide remains, while the unreduced material falls through into a lower room, where it is collected to be used again for the formation of carbide. The mixture of lime and coke is fed into the car through the flues, *e*, which extend along the whole length of the car. The rods, *f*, which bear four blades, extend through the whole breadth of the feeding flues. These rods are turned automatically, and the faster they turn the more material is fed into the car. In order to stoke the furnace automatically, the car is attached to an iron bar, *g*, by two hangers and a coupling in front of the car. Bar *g* extends through the back wall of the furnace, and is automatically moved forward and backward for about 2 inches and about twenty times per minute. The car is thus also rolled backward and forward on the track for about 2 inches each time. Every time that the car stops or starts it gets a little jerk which is sufficient to fill up the holes made by the escaping gases in the loose material. This motion of the car further prevents the arc being located for a longer

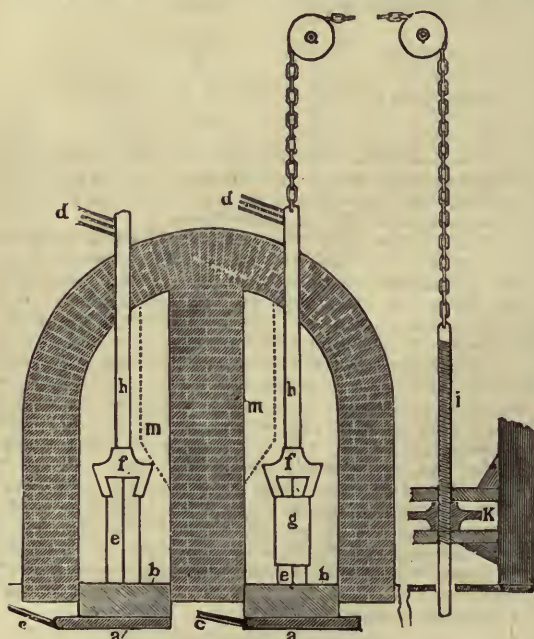


FIG. 1.

time at one point, for which the arc has always a great tendency. This will materially increase the efficient use of the heat of the arc. Under the track of the car is the bin, *h*, in which the unreduced material is collected that will fall from the car when this is taken out. This material can from time to time be taken out through the door *i*. The carbon holder is more complicated than in the Spray furnace. Twelve carbons are used, and the holder is therefore about twice as heavy. It is not advisable to suspend this carbon holder from a copper bar, which moreover becomes rather hot in this closed furnace. The carbon holder is therefore attached to a rod, *l*, which is composed of three slabs. The inner one is of copper, and measures 6 by 1½ inches, and the outer ones are of iron and are 6 inches by 1. Since it is not practical to attach the twelve carbons in their iron casing to the carbon holder in the furnace, the holder itself is composed of two pieces, *m* and *n*, which slide into each other. The aggregate of pencils is connected to piece *n* outside the furnace, and the whole is placed in the car *a*. Rod *l* is so far lowered that piece *m* will easily slide into piece *n*, and the connection can easily be effected. Iron plates, *o*,

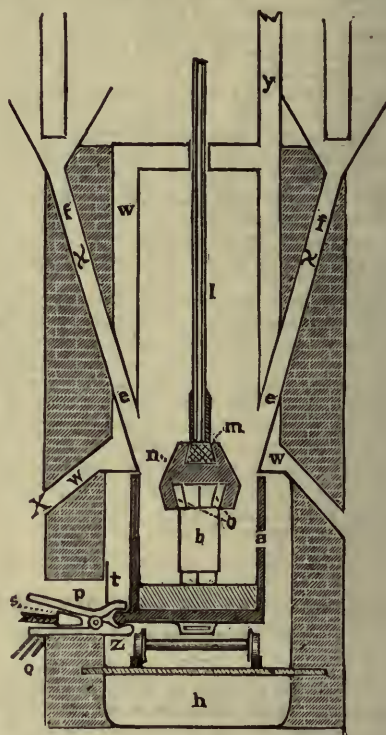


FIG. 2.

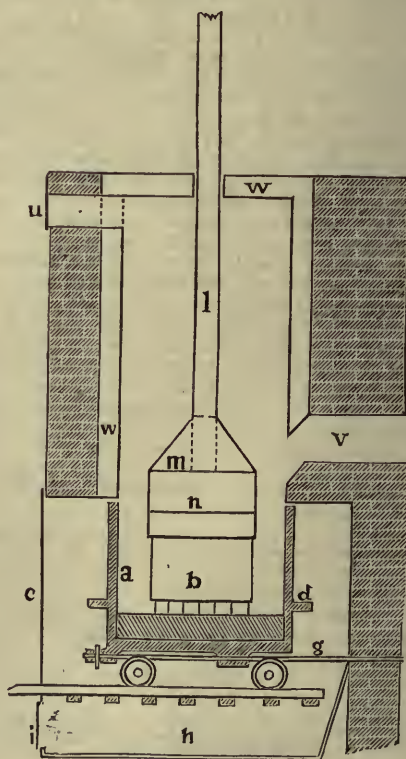


FIG. 3

are placed between the carbon holder proper and the pencils. These plates, *o*, are about 1 inch thick. They are fastened to the inside of the carbon holder by pins which are inserted in the holder, and fit in holes of the plates. These plates can be easily removed and replaced. It will sometimes happen that a small arc is started between the pencils and the inside of the carbon holder, and a part of the carbon holder will melt. In the case that the plates *o* are used one can simply replace these plates. The car *a* forms one of the electrodes, and is connected with the bottom cables, *g*, by two clamps, *p*. The lower clamp is stationary and the upper one can be opened. The clamps are tightened around the appendage *z* of the car by a wedge and screw, *s*. When the clamps are fastened the slide *t* is lowered so as to shut the opening. The electric connection with the car can also be and is better made through the bar *g*, which in that case is composed of an iron and a copper slab. It may also be made by two copper bars which run alongside of the car and are pressed against it with springs. The furnace is entirely closed. When it is started the door *c* is shut, but the door *u* is kept open till the carbon monoxide, which is formed in the reaction, has replaced the air in the furnace. This point is reached when the flame comes out of this door. Door *u* is then also closed and the gases escape through the chimney, *v*. The use of door *u* prevents explosions of the carbon monoxide in the closed furnace. Chimney *v* begins just over the car. The carbon holder and the rod *l* are therefore not in the current of the hot gases. The upper part of the furnace is cooled moreover by an air jacket, *w*, through which a draught of air is maintained. The cold air enters through openings *x* and the warm air is led off by chimney *y*. The warm air may be utilised for heating the building. The chimney gases pass through flues or rooms, in which the lime dust is collected by proper means. Owing to valuable suggestions of our superintendent, Mr. J. C. King, this furnace is called the King Furnace. Besides these two types of furnaces, several others have been proposed.

In order to start our present furnace, we shut the lower iron door and lower the pencils to the bottom of the furnace. The current is turned on and the mixture of coke and lime fed in, the arc being kept covered with the mixture as high as one foot around the pencils. It is then easier to keep the arc steady. It is necessary to stoke from time to time, for the gases which are formed in the arc constantly make channels through the material, and especially if unslacked lime is used. These channels will not fall in, and less material will come into the arc. The feeding in of the material is continued for several hours. If the attendant at the hand wheel sees that the voltage becomes low, he raises the pencils. If the arc should be broken the amperage becomes zero and the voltage high, and in that case the pencils are quickly lowered. After shutting off the current it is well to allow the furnace to cool two or three hours before emptying it.

(To be continued).

QUANTITATIVE ANALYSIS OF SPECTRA.

A NEW method has been elaborated by G. and H. Krüss, and is now published by H. Krüss after the death of G. Krüss (*Zeit. Analyt. Chemie*).

If a substance in a layer of a substance, of a thickness = *x*, enfeebls by absorption the light which it transmits from the intensity—

$$I \text{ to } \frac{I}{n}$$

(and has therefore the enfeebling factor $\frac{I}{n}$ for the stratum

1), the intensity *I'* of the light passing through a stratum of the thickness *m* will be—

$$(1) \dots I' = \frac{I}{n^m}$$

(This applies strictly for monochromatic light with only one factor of enfeeblement).

If we assume the original strength of the light = *I*, we obtain the equation—

$$(2) \dots I' = \frac{I}{n^m}$$

$$\text{Therefore } \log. n = -\frac{\log. I'}{m}$$

If we call *e* the coefficient of extinction of the substance concerned,—that is, the reciprocal value of the thickness of the stratum which is necessary to reduce the transmitted light to $\frac{1}{10}$ th of its original intensity,—then when—

$$m = \frac{1}{e} I' \text{ will} = \frac{1}{10}$$

Hence follows $\log. n = e$, or also—

$$(3) \dots e = \frac{\log. I'}{m}$$

If we work with the stratum *m* = 1 we have—

$$(4) \dots e = \log. I'$$

The coefficient of extinction is hence equal to the negative logarithm of the residual brightness. When this has been determined for the stratum = 1 it may be taken directly from the proper tables.

The authors justly point out that to many the process is rendered less intelligible by the circumstance that we do not measure the thickness of the stratum at which the brightness is reduced down to $\frac{1}{10}$, but in its stead that effected by a constant depth of stratum which is of different thickness for each body.

In order to draw a conclusion as to the concentration of a solution from its coefficient of extinction as thus determined, we must consider that the reduction of brightness depends only on the quantity of the absorbent substance present in a given depth of the solution, so that a greater concentration of the liquid at the same depth must have the same effect as the introduction of a corresponding deeper stratum of a solution of an unchanged concentration. Hence it follows that the ratio of the concentration, *c*, to the coefficient of extinction, *e*, for each absorbent substance, is a constant which must depend on the kind of the substance. It is known as the absorbed proportion—

$$(5) \dots A = \frac{c}{e},$$

whence, if *A* is known, the concentration follows from the measurement of *e*.

$$(6) \dots c = A \cdot e.$$

The authors, in order to obviate the above difficulty, have modified the procedure, so that the depth of the stratum, *m*, which occasions a certain reduction of brightness, is really measured.

If the intensity, *I*, of the light, is reduced to *I'* by a stratum, *m*, of a solution of the concentration, *c*, whilst a stratum of the thickness 1 of a solution of the same substance of the concentration 1 reduces the light to $\frac{1}{n}$, we have—

$$(7) \dots I' = \frac{I}{n m c}$$

If we put—

$$I' = \frac{I}{x},$$

then follows $x = x = n m c$, or $\log. x = m c, \log. n$.

$$(8) \quad \therefore c = \frac{x}{m \log. n}$$

n , the specific power of absorbing light, is for each substance a constant; accordingly also $\log. n$ respecting its reciprocal value, we call k . Thus we have—

$$(9) \quad \therefore c = \frac{k}{m}$$

This magnitude k is nothing better than the absorptive ratio A as above reduced, for we had above $c = A. e$, and we have here further (if m is selected so that $x = 10$),—

$$e = \frac{1}{m},$$

whence there follows—

$$c = \frac{A}{m}.$$

The apparatus used by the authors cannot be intelligibly described without the accompanying figure.

In using the apparatus we pour into one of the vertical tubes the solvent liquid, and into the other the liquid to be examined, and we allow the light from a Hübner reflecting prism to fall upon a spectro-photometer, so adjusted that, with equal illumination, one-half of the field of vision has only one-tenth the brightness of the other half.—*Zeit. Analyt. Chemie.*

CASES OF ISOMERISM IN THE PYRAZOL SERIES.

By R. VON ROTHENBURG.

1. The Question of the Constitution of the (c)-Phenylpyrazol Series.

I HAVE mentioned in the *Berichte* (xxvii., 789) that I have obtained Buchner's phenylpyrazol, melting at 228°, along with other more fusible products, by the action of hydrazin hydrate upon benzaldehyd.

Knorr, by the reciprocal action of the same substances, has obtained merely phenylpyrazol, melting at 78°. He believes, on the basis of theoretical speculations, that the phenylpyrazol melting at 228° must be (4)-phenylpyrazol, and hence declares my statement as "puzzling."

Recently, Buchner has contradicted the views of Knorr, and shown that neither his theoretical expositions nor their experimental proofs are in all respects trustworthy.

Before anew taking part in the discussion, it seems advisable to describe accurately the experiment which I formerly instituted.

Crude benzoylaldehyd—obtained in the ordinary manner—was mixed with $\frac{1}{2}$ mol. hydrazin hydrate in an alcoholic solution, producing a violent reaction. The mixture was then heated for some time in the water-bath, poured into several times its volume of water, the oil was taken up in benzene, separated from the water, and the basic constituents were withdrawn from the oil by means of dilute hydrochloric acid. The solution thus obtained yielded the platinum salt which I have described (*loc. cit.*), and after neutralising with soda a crystalline mass which, on fractional crystallisation, yielded first slightly yellowish crystals melting at 228°, and subsequently fractions fusible below 100°, which I have already described as an isomer. The yield of product fusible at 228° was about 10 per cent of the amount to be theoretically expected.

That both phenylpyrazols (melting-points 228° and 78°) should be obtainable from benzaldehyd does not agree with Knorr's theory of the pyrazol nucleus, but it does not clash with the behaviour of phenylhydrazin with benzoylaldehyd; the less so, as both pyrazols were also obtained by means of diazoacetic ester, and in various proportions,

without succeeding in exactly ascertaining the experimental conditions favourable to the formation of either isomer.

In spite of the brilliant experimental researches of Knorr on the pyrazols, which showed the formation of only one methylpyrazol from oxymethylenacetone, which allowed the same methylpyrazol to be obtained from two isomers of the phenol series, and in spite of the production of a (3, 4, 4, 5)-tetramethylpyrazol, no unobjectionable proof has been furnished that also (3)- and (5)-phenylpyrazol must be identical, since it is evident that the methyl and the phenyl-group must have a very different influence.

(5)-Phenylpyrazol, or pyrazol (5)-carbonic acid, must be obtainable by the oxidation of the (5)-phenylpyrazolin obtained by me from cinnamelydenazin. May I, since I cannot at present execute these experiments, address to Dr. Buchner the request that he would include these investigations within the scope of the oxidations which he has in view?

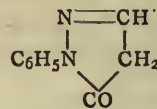
Buchner has pointed out that a resorcin fusion performed at too high a temperature is no proof for the presence of an *o*-dicarbonic acid; since resorcin alone, if heated for a long time at a sufficiently high temperature, or in presence of a condensation agent, yields strongly fluorescent melts. Knorr has totally overlooked this fact.

Buchner's resorcin fusion at a sufficiently low temperature, and my synthesis of hydrazin hydrate, require imperatively that phenylpyrazol (228°) must not be regarded as (3)- or (5)-phenylpyrazol.

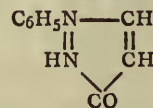
If Knorr does not view Buchner's or my statements as founded on air, he must admit that phenylpyrazol (228°) cannot be (4)-phenylpyrazol.

2. On the Synthesis of Antipyrine.

About a year ago I described (*Berichte*, xxvi., 2974) a method for obtaining a phenylpyrazol (melting-point 155°), previously obtained by different methods, and I assigned it the constitution—



On the contrary, F. Stolz advocated the formula—



He grounded his opinion essentially upon the circumstance that the isomeric phenylpyrazolon melting at 118° must have the formula which I assumed for the phenylpyrazolon melting at 155°, since it is formed from oxalacetic ester hydrazon. I did not then touch upon this point, as the production of phenylpyrazolon melting at 118° from oxalacetic ester hydrazon takes place in a manner which admits of no inference as to its constitution. On the other hand, formalacetic ester forms with phenylhydrazin not the phenylpyrazolon fusible at 118°, but that melting at 155°.

Stolz certainly contests that this pyrazolon can here come into consideration, but he has not found it necessary in any manner to establish his opinion. Why, lastly, has he not obtained (1)-phenylpyrazolon from oxalacetic ester and phenylhydrazin in the same manner in which I obtained pyrazolon itself by means of hydrazin hydrate?

In the meantime there have appeared memoirs and patent specifications which induce me to oppose energetically the constitutional formula of Stolz as entirely untenable.

If, as Stolz assumes, phenylpyrazolon fusible at 118° were really the lower homologue of Knorr's (1)-phenyl-(3)-methylpyrazolon fusible at 127°, both would be

obtainable in an analogous manner by the circuitous way of phenylethoxypyrazol; but this is not the case.

Whilst the hydrazones of the β -ketonic esters when heated alone pass into normal pyrazolones, with abscission of alcohol, they are converted by acid condensation agents into alkyloxy-pyrazols, and by concentrated sulphuric acid into indol-derivatives.

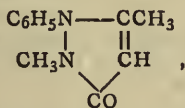
It seems to me free from doubt that the formation of indol, and that of the alkyloxy-pyrazols, occasionally occur collectively, and the non-symmetric hydrazines formed split off ammonia or water.

It is not surprising that in an acid solution the more basic pyrazol is formed rather than the very feebly basic pyrazolon. It can be explained only under these suppositions, that we do not obtain from acetacetic ester pyrazolon Knorr's pyrazolon fusible at 127° .

From the (1)-phenylpyrazolon-(3)-carbonic ester the Höchst Colour Works obtain (1)-phenyl-(2)-methylisopyrazolon of the melting-point 117° .

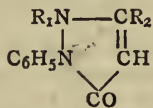
From Walker's (1)-phenylethoxypyrazol there is certainly also formed a phenylmethylisopyrazolon fusible at 117° , but it must be (2)-phenyl-(1)-methylisopyrazolon, which melts at about the same temperature. The melting-points of the (1, 2)- and (2, 1)-isomers are so near together that they cannot be used for characterising and distinction.

Knorr's pyrazolon (127°), as already remarked, is different from the "phenylmethoxypyrazol" obtained on treating the phenylhydrazon of acetacetic ester with acid condensing agents and saponifying the alkyloxy-pyrazol. Consequently, their lower homologues may be different. The pyrazolon derivative,—

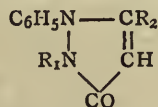


must be pseudoantipyrin.

The halogen-cretonic acid, produced according to the German patent No. 64444, is pseudoantipyrin, as Krauth has shown. This question is of no little scientific interest, since the antipyrins of the type—



are antipyretic medicines, whilst those of the type—



are poisons.

As appears from the above explanations, the elaboration of the pyrazolon region carried out by Stolz and the Höchst Colour Works is, from a scientific point of view, not unobjectionable.

The German Patent No. 66808 declares it possible to obtain pyrazolon derivatives by the oxidation of β -amido-crotonicanilidon, which is known to be impossible.

Finally, the Höchst Colour Works have not been able to refrain from announcing the notorious antipyrin alcohol for a patent, on January 5, 1892.

3. On the Pyrazolon-sulpho Acids.

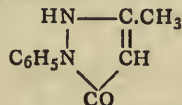
C. Walker recently describes some sulpho-acids of the pyrazolones which he has obtained by means of concentrated sulphuric acid from the hydrazones of substituted acetacetic esters. To these sulpho-acids he ascribes a peculiar and at least improbable constitution.

Walker's formulæ prove anew to what improbable assumptions the obstinate adherence of Neff and his

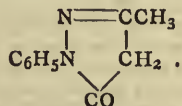
school to the oxycrotonic formula of acetacetic ester and the isoconstitution of the normal pyrazolons must lead.

4. On the Isomeric Benzoylphenylmethylpyrazolons.

Neff believes, as is well known, that he has demonstrated for (1)-phenyl-(3)-methylpyrazolon (fusible at 127°) the formula—



because he has succeeded in producing two isomeric monobenzoyl-derivatives. The proof has, however, been in no respect supplied. He overlooks that all the other reactions of this pyrazolon fusible at 127° can be much more easily explained by the formula—



How will he explain the solubility of the pyrazolons and the insolubility of antipyrin in alkali by means of his formulæ?—*Journal für Praktische Chemie*, New Series, vol. li., p. 157.

ON SUBSTITUTED GLYCOLIC ESTERS AND GLYCOLHYDRAZID.

By TH. CURTIUS and A. SCHWAN.

THIS paper forms the fifth part of a prolonged account of the hydrazides and azides of organic acids, appearing under the name of Th. Curtius.

Some years ago one of the present writers found that if benzoylglycolic ester is exposed to the action of 2 mols. ammonium hydrate benzhydrazid and amidoglycocol are produced with abscission of water and alcohol.

This remarkable reaction yielded the first representatives of two classes of organic substances hitherto unknown, the primary acyldhydrazides and the hydrazin acids.

As subsequent researches had shown that primary acyldhydrazides are quite generally formed by the action of hydrazin hydrate upon acid esters, we might have expected that benzoylglycolic ester with 1 mol. hydrazin hydrate would yield benzoylglycol hydrazid. The latter would then be resolved by the action of a second mol. of the base into benzhydrazid and glycolhydrazid.

One of the authors had previously expressed the view that the last-named substance would correspond to the constitutional formula $\text{NH}_2\text{NH}\cdot\text{CH}_2\cdot\text{COOH}$, and that therefore, in addition to benzhydrazid, there would appear hydrazinacetic acid or amidoglycocol on the scission of benzoylglycolic ester.

We have established that our view here given is correct, and that the substance in question is the hydrazid of glycolic acid, $\text{CH}_2(\text{OH})\text{CONHNH}_2$.

In the action of alcohols or acids upon diazoacetic ester one of us has found a method for producing substituted glycolic esters of any kind. By means of this process we have obtained a series of such bodies, hippurylglycolic ester, oxalylglycuric ester, succinylglycolic ester, benzylglycolic ester, and glycolic ester.

The organic acids react in some cases explosively upon diazoacetic ester even in the cold, especially oxalic acid. With other acids the reaction occurs only in heat, but then very energetically.

Pure water or alcohol reacts only with extreme difficulty upon perfectly pure diazoacetic ester, so that it has

not been practicable thus to obtain a good yield of glycol esters.

As for the effects of hydrazin hydrate upon these esters the reaction might either take place as with ordinary esters, so that glycolhydrazides are produced in which the hydrogen of the hydroxyl group is substituted by an alkyl or acetyl group, or—as the substituted glycolic esters might be regarded as double esters—there might react simultaneously, the one attacking the esterified carboxyl group, and the other the etherified hydroxyl group.

Investigation has shown that upon alkylglycolic esters there reacts only *one* mol. of hydrazin hydrate, forming alkylglycol hydrazid and splitting off alcohol, whilst in the acetyl-glycolic esters there always react *two* mols. of hydrazin hydrate.

As for the constitution of the so-called hydrazin-acetic acid we have to consider firstly its formation from glycolic ester. Hydrazin hydrate acts violently upon this substance even in the cold. On evaporation there remains hydrazid in massive crystals. But if in place of glycolic acid we employ alkylised glycolic acids, then if the substance in question is to be regarded as hydrazin acetic acid, hydrazin acetic acid should also be formed with abscission of alcohol, instead of which we always obtain alkylised glycol-hydrazid.

Finally, the remarkable instability of the so-called amidoglycocol in an aqueous solution as against dilute mineral acids, compels us to regard the compound as glycolhydrazid.

The authors then describe the preparation and properties of glycolic ester, of phenylglycolic ester, of benzylglycolic ester [$C_{11}H_{14}O_3$], of ethylglycolic ester, hipperylglycolic ester [$C_{11}H_{13}O_3N$], oxalylglycolic ethyl ester [$C_{10}H_{14}O_8$], succinylglycolic ester [$C_{12}H_{18}O_8$].

The authors next study the action of hydrazin hydrate on the substituted glycolic esters.

pear-shaped phial, covered with a watch glass with 30 to 40 c.c. of strong hydrochloric acid. A heat of 80–100° was applied by means of the water-bath or the sand-bath for fifteen to twenty hours. It was evaporated to dryness to render the silica insoluble; it was then taken up with a few c.c. of aqua-regia composed of equal volumes of nitric and hydrochloric acid. Heat was applied, and there were further added 5 c.c. of nitric acid to expel the hydrochloric acid. Lastly, it was diluted with cold water, and the whole poured into a flask graduated from 60 to 120 c.c. The volume is made up and the whole filtered through a dry filter-paper. We take 50 or 100 c.c. of the liquid (corresponding to 0.5 or 1 grm. of the substance), neutralise with ammonia, acidify slightly with nitric acid, heat to 60° in a small pear-shaped flask, and effect the precipitation by adding 20 to 30 c.c. of molybdic solution. Allow the mixture to settle for two to three hours at 30 to 40° C., filter the phosphomolybdic precipitate through a double tared filter; wash with water acidulated with nitric acid (40 c.c. acid per litre); dry the filter at 105° and weigh.

The weight of the phosphomolybdate multiplied by 0.0163 gives the weight of the phosphorus.

2. *Fusion with Alkaline Carbonates*.—Melt 0.600 grm. coal with 3 grms. of a mixture of equal weights sodium and potassium carbonates, and keep it in a state of fusion in a platinum crucible for ten to fifteen minutes. When cold the mass is taken up in water acidulated with hydrochloric acid. The liquid is poured into a porcelain capsule, adding then an excess of hydrochloric acid, and evaporating to dryness. The process is continued as in the first method.

To obtain accurate results it is necessary to fuse with alkaline carbonates and precipitate with molybdic solution according to the directions of the present paper.—*Comptes Rendus*, cxxiii., No. 23.

DETERMINATION OF PHOSPHORUS IN THE ASH OF COAL AND COKE.

By LOUIS CAMPREDON.

THE proportion of phosphorus in the ash of coal or of coke yielded on carbonisation is particularly important, when the fuel is cast into the blast-furnace, for the manufacture of fine castings in which the percentage of phosphorus has to be as low as possible. In fact, all the phosphorus introduced into the melted mass with the ash of the reductive combustible enters into the metal.

Procedures Followed.

All authorities agree in advising the determination of the phosphorus in the ash given by the combustible when burning, and not in the combustible itself. The opinion of various authors concerning the mode of attacking the ash, so as to dissolve the phosphorus contained, differs slightly.

The attack with hydrochloric acid is recommended by Fresenius, by Post, and Munk. The last mentioned adds: "There is no reason to fear that in this primitive treatment iron phosphate may remain undissolved. In the experiments made on this point the contrary has always resulted; even the residue appeared very rich in iron."

On the other hand, Blair, Baron Juptner von Jonstorff, as well as Arnold, recommend the fusion of the ash as the preferable if not the only method.

Comparative Trials.

I have made numerous comparative trials on the ash of coals from English sources, proceeding as follows:—

1. *Attack with Hydrochloric Acid*.—I treated 0.600 grm. and 1.200 grm. of ash very finely powdered in a

DERIVATIVES OF COLUMBIUM AND TANTALUM.*

By MARY ENGLE PENNINGTON.

AMONG the more metallic members of Group V. of the Periodic system are the elements columbium and tantalum, which, though almost a century old and counting among their devotees such investigators as Rose, Hermann, Marignac, Rammelsberg, and others of equal fame, still offer many interesting problems to the student of inorganic chemistry. Comparatively few of the compounds of these elements have been prepared. Those which have been studied narrowly enough to afford an accurate knowledge of their chemical behaviour form a much shorter list. The early literature is, in many instances, very contradictory, due to the supposed existence of such elements as pelopium and ilmenium, engendering as they did the fruitful controversy between Hermann and Marignac, which controversy resulted in the tacit acceptance by the chemical world of Marignac's statement, that columbium is elementary. The old doubt, however, appears to have been revived through the very careful work of Krüss in 1887, on the oxides of these metals, their separation from each other and also from the oxides which accompany them in their apparent minerals.

He found through the fractional crystallisation of the double fluoride of columbium and potassium, and by determining the atomic value of the various fractions, that something apparently contaminated the columbium. In some fractions the values obtained were far too low. This he accounted for by proving the presence of titanium.

* From the author's thesis presented to the University of Pennsylvania for the degree of Ph.D., 1895. From the *Fourth. Amer. Chem. Soc.*, xviii., January, 1896.

Other portions, however, were much too high, and this, it was carefully proved, was not due to adhering tantalum. Just what the substance was which gave in one fraction an Rv having almost double the accepted atomic mass was left undecided.

A careful consideration of this question in the light of the various researches, makes it seem not improbable that the compounds of columbium, as we know them, are not perfectly free from contaminating substances. The many difficulties encountered in the separation of this oxide from others usually occurring with it, and the insufficiency of the prevailing methods of separation, seem to demand a more exact knowledge of the behaviour of the element in the purest condition obtainable, and also when mixed with the oxides of tantalum and titanium which usually adhere to it.

It was with the hope that some additional light might be thrown upon the general deportment of the derivatives of these elements that this research was undertaken.

The material used was obtained from a columbite from Wakefield, N.H. An abundant supply of the mineral was secured through the kindness of Prof. S. P. Sharples, of Boston, in whose possession it had been for some years, though it had never been analysed. Wakefield is a new locality for columbite. The deposit was discovered while mining for feldspar. Near the columbite is quite a deposit of beryl.

Analysis of Wakefield Columbite.

The mineral occurs in large black lustreless masses. Scattered over the surface are little patches of a bright yellow substance. These proved to be uran-ochre, and gave evidence of the presence of the uranium which was later found in the mineral. Feldspar occasionally penetrated the mass, though in small quantity. The specific gravity of picked material was found to be 5.662 at 4° C.

Decomposition was effected by the method usually employed for this class of minerals.

Fusion with Acid Potassium Sulphate.—The finely divided mineral was allowed to stand over calcium chloride for some hours. The desired amount of this dry, and almost impalpable, powder was weighed off and mixed with at least nine times its weight of fused potassium bisulphate. This must be an intimate mixture. Great care should be exercised when the heat is first applied, else loss by spattering will occur. Frequent stirring tends to prevent this, and also hastens the decomposition.

Some trouble was experienced by the fusion "climbing," and leaving far up on the sides of the crucible particles of mineral which could neither be driven down by heat or forced down by a platinum rod. To collect these particles the crucible containing the clear quiet fusion was slightly tilted and the adhering portions covered with a little bisulphate. Then by gently heating the whole mass was driven down until it met the main portion of the fusion. All decompositions by this method were made in a large platinum crucible or platinum dish. The latter was preferred. If the mineral is fine enough the fusion is complete in about five hours.

The fused mass was taken up in a large quantity of water, and boiled out with water several times. The insoluble portion consisted of the oxides of columbium, tantalum, titanium, tin, tungsten, and any silica which was present. Small quantities of these oxides invariably remained dissolved, although the solution was boiled for a long time; it is, therefore, advisable to let the filtrate stand twenty-four hours, then re-filter.

The moist oxides, according to Headden (*Amer. Jour. Sci.*, xli., 91, 1891), should "be digested with yellow ammonium sulphide" to remove all tin, tungsten, &c. Rose recommends that yellow ammonium sulphide should be simply poured over them, and that this solution should be evaporated to dryness and gently ignited, to render the columbium and tantalum oxides which have been

dissolved by the alkali, insoluble. Wöhler ("Mineral Analyze," p. 140) claims that it is sufficient to treat the metallic oxides upon the filter with yellow ammonium sulphide. As some uncertainty existed as to the best course to pursue, the effect of ammonium sulphide when mixed with these oxides for a longer or shorter period of time was studied.

Heating in a porcelain dish on the water-bath for three hours gave 1.15 per cent of the mixed oxides; one and one-half days, 1.60 per cent; three days, 1.85 per cent; one week, 2.24 per cent. By pouring the sulphide over the oxides on the filter, as Rose and Wöhler advise, 0.24 per cent of the mixed oxides was obtained.

Apparently, the moist metallic oxides are more readily dissolved by ammonium sulphide than is generally supposed, and, therefore, when working with columbites containing the acid oxides, care must be taken, or a very appreciable error may result.

The ammonium sulphide solution was precipitated by dilute hydrochloric acid, and the precipitate was filtered, and washed with hydrogen sulphide water, alcohol, ether, carbon disulphide and ether. The mixed sulphides were carefully heated in the air, then reduced in a current of hydrogen gas. The residue treated with dilute hydrochloric acid gave tin in solution, and left undissolved a small quantity of a black compound which proved to be the tetroxide, Cb_2O_4 , with possibly a little tantalum.

The moist oxides when treated with ammonium sulphide have not only the acids removed, but the iron contained in them is changed to sulphide. This is dissolved out by dilute sulphuric acid. Filter off the oxides and wash them thoroughly with boiling water. A pump is usually necessary because of the precipitate being finely divided, and having a tendency to clog the pores of the filter. By this treatment the oxides should be entirely freed from iron and manganese. Nevertheless ignition gave a powder having a distinct pinkish yellow hue, showing the presence of these elements. The oxides were, therefore, re-fused with potassium bisulphate and treated as before. The second fusion gave a product lighter in colour, yet not perfectly white. Another fusion was resorted to, and no loss in weight was observed, as a small amount of iron still adhered to the oxides. In fact a perfectly white mixture of the oxides has not been obtained by this method.

The sulphuric acid solution of the iron which remained with the insoluble oxides was added to the aqueous extraction of the fusion. This solution now contained iron, manganese, uranium, and calcium, with a large excess of sulphuric acid and alkali salt. Yttrium, cerium, and calcium were looked for according to the plan presented in Rose's "Handbuch der Analytischen Chemie," ii., 335, which is, in brief, this:—The greater part of the free acid is neutralised with sodium carbonate; sodium acetate is added, so that acetic acid is in large excess. The earths are precipitated by ammonium oxalate, the precipitate being allowed to stand twenty-four hours. From 3 grms. of mineral only a very small amount was obtained. This was too small a quantity to investigate further, so that if any rare earths are present in the mineral they exist in traces.

To the filtrate which contained iron, manganese, and uranium, were added ammonium sulphide and ammonium carbonate. The iron and manganese were precipitated as sulphides, while uranium was held back by the ammonium carbonate. Beryllium, if present, would have been found here. This element was sought for, since the locality from which the mineral came made it a probable constituent, but none was detected. The sulphides having been filtered out, the filtrate was made acid with hydrochloric acid, the carbon dioxide boiled off, then the uranium precipitated by ammonium hydroxide. The uranium hydrate was filtered, washed, ignited, and weighed as U_2O_8 . The sulphides of iron and manganese were dissolved off the filter in hydrochloric acid, oxidised, and separated by the basic acetate method, the man-

ganese being finally weighed as manganese pyrophosphate.

The water contained in this columbite was determined by heating in a boat in a glass tube, and collecting the aqueous vapour in a weighed calcium chloride U-tube.

In the literature relating to columbites and allied minerals, while a ferrous content is given, the method by which it was determined is omitted. Perhaps this is due to the fact that the customary decomposition with sulphuric acid in a sealed tube naturally suggests itself, yet in applying this course to the columbite under examination unexpected difficulties were encountered. The experience is at least interesting.

To be continued).

NOTICES OF BOOKS.

On certain Main Problems of the Natural Sciences, and in particular on (I.) The Mechanical Processes which lie at the Foundation of the Electrical Phenomena. A Discourse by Dr. ANTON K. GRÜNWALD, Extraordinary Professor of Mathematics at the Imperial and Royal Technical High School of Prague. Delivered at the Annual Meeting of the Royal Bohemian Society of Sciences, January 31st, 1895. Prague: Published by the Royal Bohemian Society of Sciences. ("Ueber gewisse Haupt aufgaben der Naturwissenschaften, und zwar (I.) Ueber die Mechan Vorgänge welche den Electr. zu Grunde liegen").

PROFESSOR GRÜNWALD is already most favourably known to British physicists for his mathematical investigations on spectroscopic phenomena. We find him now taking up a much wider question. In his introductory remarks he announces that, with the present discourse, he purposes commencing a series of unassuming presentations on some main questions of the Natural Sciences. Their solution by the co-operation of experimental and theoretical research will alone render it possible for us one day to elaborate an accurate representation—satisfactory to our craving for causality—of that certainly small portion of the universe which lies within our reach. He considers that, though his present theme is *in appearance* merely and specially physical, yet in reality it extends into the weightiest questions, not merely of physics, but also of chemistry, physical astronomy, and even of physiology. His immediate subject is the mechanical procedures which lie at the basis of electrical phenomena, or, as the author puts it more cautiously and modestly, which *seem* thus to lie at the foundation.

The misfortune is that Professor Grünwald's expositions are apt to be only dimly intelligible to other than physico-mathematical specialists. Had he been able to give some brief instances showing how his views lead us into questions of chemistry and physiology, he would have reached a wider, and certainly not less appreciative, circle of disciples.

As a summary of his results, he expresses himself to the effect that the exact mathematical elaboration of the mechanical theory of electrical phenomena still requires difficult and profound study. We must especially get rid of the unsatisfactory conception of *actiones in distans*—between matter and matter on the one hand, and between matter and ether on the other—as mysterious non-mediated effects through a space supposed void and entirely without influence.

In its place must come the new conception of these phenomena opened out by Faraday, Maxwell, Helmholtz, Lord Kelvin, Hertz, Boltzmann, and other illustrious minds, who regard such phenomena merely as the consequences of a chain of regular transferences of motion—or rather of semblances of motion—passing from body to

body within a dynamic space which co-operates with them in an essential, or indeed decisive, manner.

According to the author's view, bodies are distinguished from each other, as *relatively* independent substances, by the substances of movement, and by their different position in universal dynamic space. The true independent and self-existing substance is the universe itself.

Quantitative Estimation of Sugars. With explanatory notes by Dr. ERNEST WEIN. Translated, with additions, by WILLIAM FREW, Ph.D. (Munich), Wellpark Brewery, Glasgow. London: E. & F. N. Spon. New York: Spon and Chamberlain. Pp. 128. 1896.

THE translator points out in his preface that to obtain comparable results of Fehling's procedure certain precautions are needful. He, in common with Dr. Wein, condemns the use of a fixed factor for converting either copper oxide or metallic copper into its equivalent of sugar. A common error committed is to boil the mixed solutions longer than the prescribed time. Another error is to add the cold Fehling's solution, heat and then boil for the specified time. In every case sugar solution must first be added when the Fehling's solution is in vigorous ebullition, and then continuing to boil for the specified time.

The arrangement for filtering off the precipitate of cuprous oxide in a Soxhlet tube is fully described.

Soxhlet tube filled is shown as a frontispiece. The bulk of the work consists of tables for the determination respectively of invert sugar in presence of beet-sugar, of invert sugar, of levulose and starch, and the volumetric determination of dextrose and maltose.

Dr. Wein's work in its original German form is highly approved of by practical men on the European continent, and it may be considered surprising that an English version has not appeared at an earlier date. Dr. Frew's translation only requires to be known to be highly valued.

Notes on Qualitative Analysis. Arranged for the Use of Students of the Rensselaer Polytechnic Institute. By P. MASON, Professor of Chemistry. Third Edition. Easton, Pennsylvania: Chemical Publishing Company Pp. 56. 1896.

LIKE not a few authors of elementary works on chemistry the writer admits that the world is overstocked with publications of a similar character, and makes the familiar excuse that he seeks to meet the requirements of his own classes. In the preface he informs us that it is his practice to hold a daily "quiz-class" upon points connected with laboratory work. A "quiz-class" is, we conjecture, what is known in Germany as a *repetitorium*. In the text we can find nothing objectionable, nor anything which has not been said already elsewhere.

Notes for Chemical Students. By PETER T. AUSTEN, Ph.D., F.C.S., Professor of Chemistry in the Polytechnic Institute of Brooklyn. Second Edition. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. Pp. 110. 1896.

A BOOK, we submit, rarely rises in public estimation if the author finds it necessary to tender an apology for its appearance. But Professor Austen has little occasion in this manner to crave favour, since his work is quite able to plead its own cause. We may, indeed, in some respects take exception to Dr. Austen's nomenclature. He writes "liter" for "litre." From the names of the halogens he lops off the final *e*, writing chlorin, bromin, &c. But why, not chlor, brom, &c.? Carbonous oxid and carbonic oxid are, if we may use the expression, somewhat uncomfortable synonyms for carbon monoxide and dioxide.

In a quotation apparently from Professor Schiff, the author candidly admits that we have not the slightest idea of how to bridge the gap between matter and sensation, and proceeds as if the existence of matter were an undoubted fact. He holds that "few words have been more abused than 'affinity.'"

The section on "water factors" brings under our notice a source of error which the American student encounters over and above those which we enjoy. The U.S. gallon represents only 8.3311 lbs. distilled water, whilst the British Imperial gallon weighs 10 lbs. Hence confusion and misunderstanding may arise. The author regrets that the use of the two vol. unit in chemical calculations is frequently omitted in English and American text-books.

Vest-pocket Medical Dictionary. By ALBERT H. BUCK, M.D. London: Baillière, Tindall, and Cox. Pp. 529. 1896.

THE object of this small but useful work is to explain the technical terms and abbreviations encountered in medical literature, which multiply with such uncanny rapidity as to puzzle, not merely scientific men whose specialities lie outside the medical profession, but even practitioners who have graduated prior to the last ten or twelve years. It will occasionally save gentlemen of the daily press from ludicrous mistakes. We were once staggered to read in an eminent political and literary organ, of a noted character dying from a gun-shot wound in the *peritonitis*! This little book will preserve reporters and editors from committing themselves in such a manner.

General Catalogue of the National Millenary Exhibition at Budapest, 1896. ("Catalogue Général de l'Exposition Nationale du Millénaire, Budapest, 1896"). Group XVIII. Chemical Industry. Edited by Dr. LEO LIEBERMAN.

THE total number of works here enumerated as chemical is 1253, employing 5702 men. Of these, however, 900 are engaged at the various gas-works. In the production of soap and candles there are engaged 735 hands, and in the metal trades 697. Chemical manures engage 55 persons, margarine 8, and gunpowder 3, though the powder mills are given as 8 (?). The match industry is said to be decreasing. Some of the branches of chemistry are in a flourishing state, whilst others are far from satisfying the demands of the country.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiii., No. 23, December 7, 1896.

Pleurisy in Man studied by the aid of Röntgen Rays.—Ch. Bouchard.—A purely medical paper.

Composition of the Gases evolved from the Mineral Waters of Bagnoles de l'Orme.—Ch. Bouchard and M. Desprez.—The gases would have the following composition in volumes:—Carbonic acid, 5.0; nitrogen, 90.5; argon, 4.5; helium, traces; total, 100.0. The presence of argon has been recognised in other sulphur springs. The waters of Bagnoles are not sulphuretted, but they are silicated like those of Canterets.

Fr. Landolph submits to the judgment of the Academy a memoir on the optical analysis of urine, and the exact determination of the proteids, the glucosides, and the non-fermentable saccharoidal matters.

Determination of Phosphorus in the Ash of Coal and Coke.—Louis Campredon.—(See p. 8).

Property of Discharging Electrified Conductors communicated to Gases by X Rays, by Flames, and by Electric Sparks.—Emilie Villari.—A reply to the criticism of Ed. Brany (*Comptes Rendus*, Oct. 28th last).

On Lithium Nitride.—M. Guntz.—Hydrogen, on thermo-chemical considerations, must decompose lithium nitride, as it is readily observed on heating Li_3N in a current of hydrogen.

Formation Heat of Selenic Acid and of some Seleniates.—René Metzner.—A thermo-chemical paper. On comparing the different numbers with the corresponding values for sulphuric acid they are all found lower, except the heat of hydration. Whilst sulphurous acid fixes oxygen directly, the oxidation of dissolved selenious acid is never direct.

Analysis of Industrial Copper by an Electrolytic Procedure—M. A. Holland.—This paper will be inserted in full.

On Ozone and the Phenomena of Phosphorescence.—Marius Otto.—The luminosity produced when ozone and water are in contact is due to the presence in the latter of organic matters, animal and vegetable. Most organic matters are capable of producing phenomena of phosphorescence with ozone.

New Ammunition Bread.—Ballard.—This bread, unlike the old biscuit, contains salt and yeast.

No. 24, December 14, 1896.

The Röntgen Rays applied to the Diagnoses of Pulmonary Tuberculosis.—Ch. Bouchard.—In the diseases of the thorax radioscapy gives instruction comparable in all points to that obtained by percussion.

On Selenium Anhydride.—René Metzler.—A thermo-chemical paper. The endothermic character is observed very distinctly on treating monohydrated selenic acid with phosphoric anhydride, according to the procedure by which Berthelot succeeded in preparing nitric anhydride.

Analysis of Industrial Copper of the Electrolytic Process; Determinations of Antimony, Sulphur, and Alien Metals.—A. Holland.—Arsenic and antimony are determined in the mother-liquor from which the copper has been precipitated electrolytically. Nickel and cobalt are determined electrolytically in the solution of the double sulphate and ammoniacal sulphate; iron is determined by permanganate. If the original copper was rich in silver, the copper which has been deposited upon the cone is dissolved, as it contains all the silver. In the contrary case a fresh portion of the copper is dissolved in nitric acid, and the nitric solution—filtered if needful—is with a chloride. The lead is determined in a fresh portion of the original substance, and is dissolved in dilute nitric acid. The sulphur is determined as barium sulphate.

Antimono-tungstic Compounds.—L. A. Hallopeau.—Antimonic hydrate can combine with tungstic acid to form antimoni-tungstic compounds, comparable to the phospho-tungstic and arsenio-tungstic combinations.

Researches on Cobalt and Nickel Sulphides.—G. Chasneau.—It results, from the author's experiments, that the alkaline polysulphides, if saturated in the cold with sulphur, give in cobaltous salts a black persulphide, Co_2S_7 , insoluble in alkaline monosulphides, but soluble, on the contrary, in these sulphides if saturated with sulphur. Under the same conditions the nickelous salts give a black sulphide, which appears to correspond with that of cobalt, but which, contrary to the latter, is scarcely soluble in sodium polysulphide, but decidedly soluble in the monosulphide.

New Process for the Determination of Glycerin.—F. Bourdas and Sig. de Raczowski.—This paper will be inserted in full.

MISCELLANEOUS.

Society of Public Analysts.—The Annual Meeting of the Society will take place on Wednesday, January 13, 1897, at the Chemical Society's Rooms, Burlington House, Piccadilly, at 5 o'clock p.m. The Year's Accounts will be presented. The retiring President will deliver his Annual Address. The following Papers are announced:—"Some Analyses of Water from an Oyster Fishery," "Note on Weighing Out Fats," "Remarks on Formaldehyde," by Chas. E. Cassal; "A Specific Gravity Pipette," by W. F. Keating Stock; "A Modified Schmidt Process," by R. W. Woosnam.

MEETINGS FOR THE WEEK.

MONDAY, Jan. 4th.—Society of Chemical Industry, 8. "Smelting and Refining of Cyanide Bullion," by A. Caldecott, B.A. "Industrial Use of a Recording Pyrometer," by Prof. Roberts-Austen, C.B., F.R.S.
TUESDAY, 5th. } Royal Institution, 3. (Christmas Lectures, 1896-7).
THURSDAY, 7th. } "Visible and Invisible Light," by Prof. Silvanus
SATURDAY, 9th. } P. Thompson, F.R.S., &c.

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1937.

THE ESTIMATION OF MANGANESE IN SPIEGELS, &c.

By H. BREARLEY.

THE acetate method of separating iron and manganese is very old. Its surname, whatever it may have been, is little heard of now. This is rather complimentary, because the much used methods are the more prone to suffer this change. Amid the quick young methods it seems to have lost virtues it was once credited with. There is more than a rumour, too, that its consort, precipitating with bromine and ammonia, and weighing as Mn_3O_4 , is not so reliable as it was formerly supposed to be.

Experience of the method in this laboratory has always been very satisfactory; but that opinion is by no means unanimous may be seen from the following references.

Blair ("Chemical Analysis of Iron," Second Edition, p. 108) distrusts the Mn_3O_4 , and recommends weighing as $Mn_2P_2O_7$. A similar opinion is expressed in Crookes's "Select Methods" (Third Edition, p. 191). Fresenius ("Quantitative Analysis," Seventh Edition, p. 205) says that accurate results cannot be got with pyrophosphate unless the Mn is re-determined in the filtrate and washings; whilst the oxides, ignited out of contact with reducing gases, are finally converted with Mn_3O_4 , whose weight remains unaltered. Arnold ("Steel Works Analysis," p. 81) says the ignition should be made in a hot muffle furnace, or the residue may not strictly conform to the formulæ Mn_3O_4 , which, under the above conditions, it will possess. Reddrop and Ramage (*Four. Chem. Soc.*, 1895, p. 268) show that the ordinary gravimetric method agrees with Pattinson's and the new one they describe.

Great stress has been laid on the evidence afforded by Pickering's researches (*CHEM. NEWS*, xliii., 226). McKenna (*CHEM. NEWS*, lxi., 184) objects to Mn_3O_4 on the evidence afforded by these papers, but later shows that ammonium manganous phosphate is soluble whether washed with water, ammonium nitrate, or dilute ammonia. Pickering himself recognises that his results are probably vitiated by the permeability of the platinum basins and consequent reducing action of the gases. The experiments of Morse and Burton (*CHEM. NEWS*, lxvii., 175) made to test the point, furnish evidence in the same direction. It is plain that changes might take place in an open platinum basin, over a Bunsen flame, which could not happen in a muffle at full red heat. Under the latter conditions concordant results are always obtained.

The general instructions for separating Fe and Mn are: iron in ferric state, cold solution, neutral or thereabouts, and an excess of acetate. In case of spiegels and high manganese alloys, the acetate precipitate is always re-dissolved and re-precipitated to ensure a complete separation. It was thought that the modification applied to iron and nickel (*CHEM. NEWS*, lxxiv., 16) might apply also to iron and manganese, and thus obviate the need for a second precipitation.

The excess of acetate necessary is variously stated "more than sufficient excess to change all the iron and manganese by double decomposition to neutral acetates" (Crookes). 30 c.c. ammonium acetate* when half-a-gram of spiegel is operated on" (Arnold). "Two grms.

sodium acetate (crystallised) to precipitate 1 grm. of iron" (Blair).

To show the effect of free acetic acid 5 grms. of spiegel were dissolved and divided into five lots, neutralised, made up to nearly 1000 c.c. with cold water, 60 c.c. ammonium acetate added to each, and boiled. The hot solution was made up to a litre, cheesed,* 500 c.c. supernatant liquid filtered off, precipitated, ignited to constant weight, &c. The temperature at which the iron solutions became turbid was noted first at slight, and second, at decided turbidity. A "standard" sample was estimated at the same time by precipitating, washing, re-precipitating, &c., and gave first separation 21.616 per cent; second separation, 0.389 per cent; third separation, 0.063 per cent; total, 22.068 per cent.

The results of the five test estimations are collected in the following table:—

No.	Acetic acid.	Per cent manganese.	Temperature of turbidity.
I.	0 c.c.	20.66	56-60° C
II.	10 "	21.14	63-65 "
III.	20 "	21.88	65-67 "
IV.	40 "	22.02	72-74 "
V.	70 "	22.06	80-82 "

The Fe_2O_3 , along with the Mn_3O_4 , was certainly less than 0.0002 (except in V.), and this mostly due to a trace of iron in the bromine. The filtrates from the basic acetates were all clear; the precipitates and excess of solution were left over-night, and it was found that the precipitates were bulky inversely as the acid addition; I. was about three times as large as V., the others were intermediate. The supernatant liquids of the I., II., and III. contained no iron, IV. a trace, and V. a decided quantity. The precipitates also varied in colour. I propose to examine these physical properties more closely.

To wash the precipitated MnO_2 when it has been transferred to the filter "till free from ammonia salts" or "thoroughly" is very troublesome, and sometimes impossible on account of the tendency it has to run through the filter as the ammonia salts disappear. There seems no reason why it should be washed at all in ordinary cases, since all the ammonia salts are volatile. Determinations with and without washing give closely agreeing results, and two filtrates (about 800 c.c.) evaporated to dryness and ignited gave 0.0168 and 0.0134 grm. residue, which was mostly SiO_2 . Not more than 10 c.c. of this solution can be in the precipitate, and not more than 5 c.c. need be if it is thoroughly aspirated, so that any error introduced is insignificant, for technical work at any rate. This, of course, does not apply when sodium acetate is used or the spiegel contains unusual impurities.

Some corrections which must be made when an aliquot part of a hot solution is taken, as above, will be mentioned later.

The great difference between the avidities of nitric and hydrochloric and acetic acid suggested the idea of adding acetic acid to the freshly dissolved spiegel, and then as much dilute ammonia as would neutralise the free HNO_3 and HCl , form the required amount of acetate and leave sufficient free acetic acid. The details need not be given, since the matter is curious rather than serviceable. After everything had been added the solution was dark coloured, but quite clear. The percentage found on the sample previously mentioned was 21.93. A fortnight later two others in the same way gave 21.92 and 22.05 per cent.

These modifications shorten the ordinary method with its re-precipitations, without in any way impairing its accuracy. But there is still the tedium of weighing and re-weighing, and the contamination of the Mn_3O_4 and Cu, Ni, Co, &c., in case they are present in the sample.

* Ammonium acetate used throughout refers to liquid acetic acid (33 per cent), neutralised with liquid ammonia.

* "Cheesed" means to wrap a duster round the beaker, and then fold another over the cover to prevent cooling.

I am not aware that Guyard's method of estimating manganese has ever been applied to the acetate filtrate. Of this method Sutton ("Volumetric Analysis," Fifth Edition, p. 190) says "Nickel, cobalt, zinc, alumina in moderate quantity are of no consequence. The method is easy of execution, and gives good results in cases where it can be properly applied, but such instances are few."

Hopefully the filtrate was nearly neutralised with ammonia, and the standard permanganate run in. The end reaction was fugitive, and the results at best were only fair. If a pink colour was produced, the precipitate at once filtered off, solution acidified in H_2SO_4 , and excess of permanganate determined with $FeSO_4$, the results were always too high; and if filtered and allowed to stand the colour gradually disappeared, and MnO_2 was precipitated. This reaction continued long after all the manganese was precipitated, but was less decided in cold than hot solutions. It was found finally to be due to the ammonium acetate and other ammonium salts. This point was much elucidated later by reading that "ammonia boiled with a neutral solution of potassium permanganate was converted into potassium nitrate" (Tamm, CHEM. NEWS, xxv., 26).

The more acetate, the greater discrepancy; so that with as little as was necessary both of acetate and acetic acid the disturbing reaction might be inappreciable in the cold. But how little acetate and acid could be used to effect a perfect separation with one precipitation?

Experiment showed that 10 c.c. of ammonia acetate would precipitate 1 grm. of bar iron in 1 litre of solution. The supernatant solution was slightly turbid, but filtered crystal clear through asbestos. 20 c.c. of acetic acid with the same acetate did not prevent precipitation, but the solution needed twice filtering to make it crystal clear.

A series of estimations made are given in the following table. It shows the effect of varying the acetate and acid under constant conditions, thus answering the question at issue, and also supplementing the table given above.

Solutions of bar iron and $MnCl_2$ were mixed to approximate a 20 per cent spiegel. The solution was made up anew for each vertical column, as available leisure did not admit of consecutive work on the series, the variation between column and column is probably due to this cause. To support this view the samples marked with an asterisk in columns 10 and 60 were made on the same solution and side by side.

Acetate acid.	Ammonium Acetate,			
	10 c.c.	15 c.c.	30 c.c.	60 c.c.
0 c.c.	20'48*	20'29	20'00	19'50
5 "	—	20'35	—	—
10 "	—	20'37	20'38	19'74
20 "	—	20'35	20'46	19'91
40 "	—	—	20'48	20'19
60 "	—	—	—	20'18
				20'47*

If we assume that each perfect separation—the one with most acid in—represents an even 20 per cent, and calculate the other results proportionately, the influence of acid and acetate is more readily seen.

Acetic acid.	Ammonium Acetate.			
	10.	15.	30.	60.
0	20'0	19'94	19'52	19'33
5	—	20'00	—	—
10	—	20'02	19'90	19'56
20	—	20'00	19'98	19'73
40	—	—	20'00	20'01
60	—	—	—	20'00

Keeping the ammonia salts as low as possible, and precipitating with only 10 c.c. of acetate, and titrating with

Wright and Menke's (*Your. Chem. Soc.*, 1880) modification of Guyard's method, some very good results were obtained.

Soda acetate in minimum quantity with soda salts behaves well in either hot or cold solutions. Half-a-grm. of crystals, after neutralising 1 grm. bar iron with sodium carbonate, brought down a precipitate, but left a supernatant yellowish liquid no amount of filtering would decolourise. Three-quarter grm. of crystals gave a clear filtrate, and the tint imparted by two drops of N/10 $KMnO_4$ was not appreciably affected when the solution was heated to boiling. The difficulty might have been eliminated at once by using soda salts, but it was thought desirable to retain ammonia if possible, so that the gravimetric or volumetric methods might be applied to the filtrate at will.

Briefly the method is—Dissolve spiegel in HCl , oxidise the iron with HNO_3 , neutralise with soda or ammonia, and precipitate Fe with acetate (sodium or ammonium) in minimum quantity. Nearly neutralise filtrate, and titrate with permanganate. This outline is given so that there may be something to definitely refer to. The method of titration preferred is Wright and Menke's in cold solutions. This is better than titrating in hot solutions because the $KMnO_4$ is less likely to be affected by accidental or unavoidable organic matter, &c. This is strongly emphasised by my experience. No distilled water was handy, our ordinary tap water was used, and titration performed in hot solutions. The results were very unsatisfactory. Of course, ordinary water ought not to have been used, but such a contingency in actual work is not unimaginable.

The following mixtures of iron and manganese chloride solutions, mixed to approximate spiegels and ferro-manganese, show that the method gives exceedingly accurate results.

Per cent manganese Present.	Found.	N/10 $KMnO_4$ (approx.) required by—		Salts used.
		Theory.	Sample.	
10	10'09	30'03 c.c.	30'3	Soda.
10	9'99	30'03 "	30'0	Ammonia
20	19'99	60'06 "	59'96	Soda
20	19'99	60'06 "	59'96	Ammonia
40	40'00	89'5 "	89'5	Soda
60	59'87	89'5 "	89'3	Soda

I am obliged to Mr. R. L. Leffler, the chemist in this laboratory, for making a trial of the method. I furnished him with an account of the method and some solutions marked with the approximate composition. He reported as follows:—

Given as (per cent) . . . 12'00 23'00 40'00 65'00 82'00
Found (per cent) . . . 10'76 21'99 40'16 64'40 79'78
They really contained (p.c.) 10'80 22'00 40'00 64'00 80'00

Soda salts were used throughout.

When so small a quantity of acetate is used, the iron solution must be neutralised pretty accurately, or it will not be clearly precipitated. A trouble of this kind might be obviated by adding soda carbonate to slight turbidity, and dissolve in, say, 5 acetic acid. The influence of this acetic, even when larger amounts of soda acetate are used, is seen from the following amounts of permanganate used.

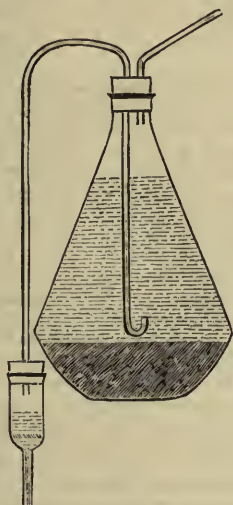
Acetic acid.	Soda acetate (20 c.c. = $\frac{1}{2}$ grm. crystals).			
	20.	30.	40.	60 c.c.
0 c.c.	59'5	58'9	58'1	56'5 c.c.
5 "	..	59'9	59'85	59'4 "

The quantity theoretically required was 59'75.

In such cases as the foregoing, where an aliquot part of a hot solution containing a precipitate is filtered off, there are certain corrections need making. Say the total bulk of solution and precipitate is 1000 c.c., and 500 c.c. of the clear solution is filtered off. From this 500 c.c. there must be subtracted (1) half the volume of the pre-

precipitate; (2) the volume contracted from the temperature when 1000 c.c. is measured to the temperature when 500 c.c. is measured; (3) a slight correction due to the concentration of the solution while filtering, &c. In case of spiegels the volume correction for bulk of precipitate is less than $\frac{1}{2}$ c.c., so of no moment. The correction for contraction was carefully considered. The most convenient way seemed to be to note the temperature in the litre flask, the temperature in the half litre, and calculate the contraction due to cooling. To this end the contraction of an ordinary 20 per cent spiegel filtrate was observed, and found to behave so much like water that Kopp's tables for that body have since been used. In some scores of cases observed the temperatures have not varied more than 2° C. under uniform conditions of working. It is not easy to fix a value for the third error. It depends on the form of vessel used, the rapidity of filtration, and other incidents which vary in an indeterminable way. Arnold ("Steel Works Analysis," p. 82) allows 5 c.c. when the volume of solution is 600 c.c., and later only 1 c.c. when the volume is 300 c.c. These also include the volume of the precipitate.

It were best to avoid this error altogether if possible. The following means were taken to minimise, if not altogether eliminate it:—The precipitation was made in flasks, Taylor pattern, marked at 1000° C.; the boiling solution was transferred to a litre flask with graduated neck, the volume noted, and the litre inverted in the precipitating flask. The precipitate readily settled, and then the clear solution was filtered by the arrangement shown in the figure. The filtration is very rapid, because little or no precipitate falls on the filter.



If small quantities of suspended precipitate made no difference to the subsequent titration, the filter tube might be dispensed with, and half a litre simply syphoned off. Results of experiments in which the iron was introduced as an emulsion of the precipitated basic acetate were—

	Grm.	Grm.	Grm.	Grm.
Fe present ..	0.0000	0.0007	0.0033	0.0050
	C.c.	C.c.	C.c.	C.c.
KMnO ₄ used..	31.75	31.8	31.75	31.6

Throughout these experiments the precipitated MnO₂ was frequently dissolved in ferrous am. sulphate, and the excess titrated with standard bichromate. This is an obvious way of confirming an estimation.

One other point, the standardisation of the permanganate solution, needs to be carefully noted. Simple standardisation with bar iron or flower wire, dissolved with precaution in H₂SO₄, has always given a value 1 or 2 per

cent below what we had good reason to suppose was the real percentage. McD. Irby (CHEMICAL NEWS, xxx., 142) has shown that the small amounts of carbon present in the iron has an effect amounting to between 1 and 2 per cent, so that the iron solution needs to be oxidised to destroy the carbonaceous matter, then reduced and the titration performed. As this point seems to be generally overlooked, and as it introduces an error of several tenths per cent on a 20 per cent spiegel, it is being studied in connection with a series of experiments on Särnström's method of estimating manganese. Up to now we have found it convenient and accurate to standardise with a spiegel containing a known percentage of manganese, or with a manganese salt whose purity was beyond question. The value of the last lot of permanganate determined by three separate spiegels was—

1.	0.001662 Mn per 1 c.c.
2.	0.001667 "
3.	0.001664 "
By pure manganese salt	0.001664 "

Conclusion.

After the gravimetric part of this paper was done, I was under the impression that separating iron and manganese in presence of considerable quantities of acetic acid was a new idea; and the same may be said of precipitating with minimum amounts of acetate. I think I will make this paper more complete than otherwise if I give the references of papers that have come to my notice.

Debray (CHEMICAL NEWS, xxi., 53).—"The acetate of sesquioxide of iron separated, on boiling, into acetic acid and colloidal oxide. . . . Colloidal oxide of iron is insoluble in ammonia salts, even in presence of a large quantity of acetic acid."

Jewett (CHEMICAL NEWS, xl., 273) shows that, with large quantities of soda acetate, acetic acid—amounting to 5 per cent of the volume of solution—causes the complete separation of Zn and Mn from iron, and a nearly complete separation in case of Ni and Co. On the authority of E. H. Smith, the same paper states that iron could not be precipitated, in presence of large amounts of acetic acid, by increasing the soda acetate. My observations are contrary to this. The error may be seen by precipitating, in presence of 70 c.c. acetic acid, with minimum acetate and several grms.

Kessler (CHEMICAL NEWS, xxvii., 14).—"The loss experienced in the estimation of the manganese by the acetate (soda) method is mainly due to the fact that too large a quantity of acetate of soda is employed, in consequence of which a portion of chloride of manganese is converted into acetate, which salts is readily converted into protoxide and acid."

I am sorry to have unknowingly gone over previously trodden ground, but the persistent way in which the instructions to use unnecessarily large amounts of acetate and neutralise all free acid are repeated, even in the most recent text-books, makes it perhaps not altogether inopportune that this matter should be again brought forward. It is plain that the results here set forth point to a modification of the method previously proposed for nickel and iron separation, in the interest of economy, if not accuracy.

Mr. Leffler suggests that the volumetric method be applied to steels. For steels of the ordinary class it offers no advantages over a number of already well-known methods, such as Ford and Williams'. In those cases, where several percents of chromium are present, it may prove useful, since such steels are frequently not completely soluble in nitric acid.

The various points enumerated are here collected into

The Method.

Dissolve 1 to 1.5 grms. of the spiegel (20 per cent) or proportionate amounts of other manganiferous irons, in

hydrochloric acid, and oxidise the iron with nitric acid. If the silica present is likely to interfere with the neutralisation, pass through a small asbestos filter. Neutralise with soda carbonate, dilute to about 900 c.c., and add soda acetate at the rate of 20 c.c. per 1 gm. of iron. Ammonia and ammonia acetate may be used, bearing in mind its limitations. See that the boiling solution touches the litre mark on the flask. Transfer to the litre measure with graduated neck. Note temperature and volume, and replace in the precipitating flask. Cheese, syphon, or filter off half the noted volume, and take the temperature. Cool, destroy the free acid with soda carbonate, and then make slightly acid with acetic or dilute sulphuric. Run the amount of permanganate into a flask, and add 10 c.c. ZnSO_4 , and run in the manganese solution with constant shaking. This may readily be done by placing a wide-stem funnel in the larger flask, holding it in position with the first finger of the right hand; the lower fingers grasp the neck with the flask resting against the thick of the hand; the solution is shaken, while the left hand pours the manganese solution through the nearly stationary funnel. The percentage of manganese in spiegel is generally approximately known. Failing this an approximation may be speedily obtained by syphoning off an additional 250 c.c. and precipitating hot (Guyard).

The well-shaken liquid containing the small excess of permanganate is allowed to settle a few minutes, an aliquot part filtered off, acidified, and determined with ferrous ammonia sulphate and permanganate.

After correcting the figures for volume contraction the calculations need no further explanation.

The following values, from Kopp's tables for water, given here, will save further reference.

If the volume of water = 1 at 0°C. , it changes when heated to the following volumes:—

70°C. ,	1'02225;	85°C. ,	1'03189;
75°C.	1'02544;	90°C.	1'03540;
80°C.	1'02858;	95°C.	1'03909.

Reagents required:—

Potassa permanganate	3'156	grms. per litre.
Soda acetate	37'5	"
Zinc sulphate	200	"

Laboratory, Norfolk Works, Sheffield.

THE MANUFACTURE OF CALCIUM CARBIDE.*

By J. T. MOREHEAD and G. de CHALMOT.

(Continued from p. 5).

THE carbide is always found in one piece between the pencils and the bottom. It has a conical form, being broader at the base, and can be $2\frac{1}{2}$ feet high in our furnace. It, however, never has so great a diameter as to fill up the whole capacity of the furnace. The carbide is therefore entirely surrounded by a cover of the mixture of lime and coke. This mixture is so bad a conductor of heat that the brick walls of the furnace are not attacked. It is very easy to separate the carbide from the loose mixture, for the latter never melts together, while the carbide is hard and solid. The pieces of carbide are covered with a thin coating which is a little thicker at the top of the piece, and the same may be re-ground and again used. This coating contains mainly carbon, but also carbide and calcium oxide. It seldom yields more than half a cubic foot of gas per pound, but in some cases it yields 1'77 and even 2'10 cubic feet. This coating, however, is of little importance. If the mixture is well made this coating seldom exceeds from twenty to thirty pounds on

a piece of carbide of from 300 to 400 pounds. The carbide itself is crystalline. The crystals are especially well developed near the top, and are more perfect with an excess of coke, low voltage, and when allowed to cool slowly. The centre of the piece of carbide stays liquid for some time after the electric current has been shut off. The liquid part, however, is of the same quality as the rest of the piece. We have, in fact, tapped out of the furnace carbide which was very pure and yielded 5'59 cubic feet of gas per pound. We do not wish to express an opinion as to the practicability of tapping the carbide as soon as it is formed. We will only mention that Mr. Price, in Newark, has, with a view of tapping the carbide, constructed and patented a new furnace, and that one of us (C.) has also devised a furnace for the same purpose.

Carbide of average quality (about 5 cubic feet of gas per pound) often has a reddish colour, especially if it has been made with a current of high voltage. Carbide of bad quality is often greyish or blackish, or will show streaks of graphite. Pure carbide yields more than 5'90 cubic feet of gas per pound. It has, however, been found to be more economical to produce carbide that yields only about 5 cubic feet of gas per pound. Samples of carbide of different qualities contained:—

TABLE I.				
Cubic feet gas per pound.	Carbide. Per cent.	Free calcium oxide. Per cent.	Carbon Per cent.	Other im- purities. Per cent.
5'7	96'6	0'6	—	2'8
5'5	93'2	4'2	—	2'6
5'1	86'4	9'5	—	4'1
5'025	84'7	10'7	1'6	3'0
3'6	61'0	27'5	3'2	8'3
3'45	58'5	1'1	25'6	14'8

The upper part of a piece of carbide is often purer than the under part.

The coke to be used should not contain much ash. Our coke contains about 7 per cent of ash. The carbide obtained with a coke of from 10 to 11 per cent of ash was perceptibly inferior to that obtained with our usual coke. It was found impracticable to make a good quality of carbide with a coke of 27 per cent ash. It is well that there should not be more than 10 per cent of ash in the coke. The coke should be ground very fine, and it should pass through a fifty-mesh sieve. The lime need not be as fine as the coke. The largest pieces should pass through a ten-mesh sieve. If the lime is coarser the quality of the carbide becomes inferior. That the state of the pulverisation of the lime is important can be seen by a comparison of the average amount of gas per pound (4'97 cubic feet), obtained with unslacked lime (Table II.), and that obtained with air slacked lime (5'27 cubic feet, Table III.). The unslacked lime was in several instances not quite as fine as the slacked lime. Unslacked lime is decidedly preferable to air slacked lime, as we will see afterwards.

The lime which we use contains $1\frac{1}{2}$ per cent magnesia and 1 per cent of other impurities. The anhydrous lime should contain 95 per cent calcium oxide and no more than 5 per cent impurities. The presence of magnesia is especially detrimental to the production of carbide. We could not obtain a good quality of carbide with a lime in the following analysis:—Insoluble, 0'24 per cent; silica, 0'78 per cent; ferric oxide and alumina, 0'68 per cent; calcium oxide, 92'83 per cent; magnesium oxide 5'47 per cent. Further experiments showed that $2\frac{1}{2}$ per cent of magnesia in the mixture has a marked influence on the production. The lime used for making carbide should not contain over 3 per cent of magnesia. That magnesia has such a bad influence upon the formation of carbide is probably due to its forming a veil between the carbon and the lime particles, preventing their combination. Magnesia does not unite either with lime or with carbon. The latter fact was first shown by Moissan (*Compt. Rend.*,

* Read Sept. 3rd before the Springfield meeting of the A.A.A.S. by one of us (M.). We have made since then several additions, so as to make the article complete up to the present time. From the *Journal of the American Chemical Society*, April, 1896.

118, 506), and our own experiments in this line fully confirm his results. The lime and the coke must be mixed very well or the carbide will be of inferior quality, and there will be much coating. Besides the carbide some mixture remains in the furnace. More carbon than lime burns out or volatilises in an open furnace. It is therefore necessary to add carbon to this mixture before using it again. The amount to be added is calculated from the result of an analysis of the mixture. If coke is added in the proper proportions the unsmelted portion of the material can be returned at least three times into the furnace and still yield good carbide. The impurities of the lime and the coke ashes remain as well in the carbide as in the residual mixture. It is therefore a good practice to add charcoal instead of coke to the mixture, so as not perceptibly to increase the amount of ash. The mixture that comes from the furnace is red hot, and it will stay hot for days. It will lose a large amount of carbon if allowed to lay in heaps in the air. It is better to mix in the necessary amount of carbon and use the mixture at once again. One can also keep the mixture in air-tight sheet-iron tanks. If the lime has been unslacked the mixture cools much quicker and does not lose as much carbon after it has been taken from the furnace. In the case of slacked lime water gas is probably formed in large amounts. The carbon pencils must be well cared for in order that they last for a long time. If sufficient coke is put in the mixture they are not attacked much at the end. They will shorten from 0.05 to 0.10 inch for every hour running. They become thinner for being exposed to the air when hot. They are mainly attacked after the electric current has been shut off, for if the furnace is working the gases from the arc come up around the carbons and shut the air off. In order to save the carbons best it is therefore well to keep the furnaces running with as little interruption as possible. In the closed furnace, which we have described, the carbons will be surrounded by non-oxidising gases, which will save them materially. In the open furnaces in Spray we surround the carbons with a sheet-iron cover that reaches from the carbon holder to within four inches of the bottom end of the carbons. This jacket is fastened with iron wires to the carbon holder. The space between the carbons and the jacket is packed with a mixture of coke and coal tar or pitch. This mixture is baked by surrounding the carbons and jacket with the red hot material that comes from the furnace, or by placing them in a fire. The jacket will generally last as long as the carbons. One set of the carbons in an open furnace, and with interrupted operations, will last on an average about 100 hours. These figures hold good where a current of from 1700 to 2000 amperes is used. The voltage has no perceptible influence on the result. Working with say 1700 amperes and 100 volts, and generating about 225 horse power, the production of carbide per hour can be reckoned to be easily eighty-five pounds, and one set of carbons can therefore make at least 8500 pounds of carbide, even in an open furnace. If the furnace is used continuously the carbons will last at least from 200 to 300 hours, and the cost of pencils for one ton of carbide will be about 1 dol.

The analytical part of our work has been very simple. After the piece of carbide has been broken open with a hammer, two or more samples, representing as nearly as possible the average quality of the carbide, and of about eight ounces each, are taken. These samples are broken in pieces of about half an inch in diameter, and from two to three ounces are taken for one gas test. The material is put into a dry bottle of about one quart capacity, which is provided with a rubber stopper, through which two glass tubes pass. The one tube bears a stop cock and drop funnel, the other tube conducts the gas through a series of U tubes and then through a small gas meter. The funnel is filled with water, and, by opening the stop-cock, water is allowed to drop slowly on the carbide. The acetylene gas is generated and is cooled in the U tubes before it passes to the gas-meter. Much water

vapour is condensed in the U tubes, for the gases generated in the bottle are hot. We make a correction for the temperature of the gas as it passes the gas-meter. We do not take into consideration the small amount of gas which passes through the gas meter by the expansion of the gas in the bottle when the latter becomes hot, and because a part of the bottle becomes filled with water. The error arising herefrom is of no consequence, for the volume of the bottle is only one quart, and the volume of the gas which passes from the gas meter is from $\frac{1}{4}$ cubic foot to 1 cubic foot. The water, moreover, becomes saturated with acetylene. Our figures show the amount of moist gas at the temperature of 60° F.

In order to determine the lime in the mixture, two and five-tenths grms. are boiled with a slight excess of hydrochloric acid of known strength in a 250 c.c. bottle. The bottle is cooled and filled up. The liquid is filtered, and in 50 c.c. of the filtrate the excess of acid is determined by titration. The coke is determined by boiling 2 grms. of mixture with 25 c.c. of 12 per cent hydrochloric acid and filtering off the coke on a Gooch crucible. These methods do not make a claim to absolute accuracy, but they can be quickly executed and give a good estimate of the relation in which the coke and lime are present in the mixture, as the following figures show. The coke used for the original mixture contained 7.33 per cent of ash. The coke that remained from the mixture that had been boiled with 12 per cent hydrochloric acid contained 6.2 per cent of ash, and the coke which remained by the same treatment from a similar mixture that had been once in the furnace contained 7.2 per cent ash. The amount of lime found in mixtures by titration, and that found by gravimetric analysis, varied only by from $\frac{1}{4}$ to $\frac{3}{4}$ per cent when the small amount of magnesia in the lime was known and taken into consideration. In controlling the different runs we have proceeded as follows:—

The carbide was weighed and the coating on it determined either by taking it off and weighing it or by estimating it on small and clean pieces. By deducting the weight of the coating from the weight of the piece of carbide we obtain the net yield of carbide. The gas therein is determined, the figure accepted being the average of the result of the analyses of at least two samples. In order to determine the power used, we multiply the voltage by the ampère and divide the product by 746 to obtain the number of horse power generated by the dynamos. In order to make a more proper comparison we found it necessary to deduct the loss of voltage sustained in the carbon pencils. Our pencils were made in different factories and had a different resistance. We therefore determined the difference in voltage as indicated by the usual reading of our meter and the voltage at the end of the carbon pencils. We touch the end of each pencil alternately with an iron rod that is connected with the volt meter by a copper wire. We call net power the power generated by the dynamos less the average loss in the six carbon pencils. Our meters are placed in the primary circuit and we have not taken into account the losses of ampère in the transformers and those sustained by leakage. We have further found that the readings of our meters are about 6 per cent higher than those of standard Weston meters. It may therefore be safely relied upon that all our estimates for the production of carbide per horse power are too low. The error is, however, in all cases in the same direction, so that it cannot have materially influenced our deductions, which are based upon a comparison of our results.

In the carbide there is also a considerable loss of voltage, and therefore of power. We found, for example, sixty-five volts in the bottom cables and only fifty volts at the top of a 2½ feet high piece of carbide just under the arc. This makes a loss of six volts for each foot of carbide. The average production during six to eight hours of continuous working is as large as that during two or three hours at the same power. It is, however, not ad-

visible to make the carbide pieces higher than $2\frac{1}{2}$ feet, since then the resistance of the carbide will begin to materially reduce the quantity of the production.

(To be continued).

DERIVATIVES OF COLUMBIUM AND TANTALUM.*

By MARY ENGLE PENNINGTON.

(Continued from p. 10.)

THE mineral was ground very fine and heated in sealed tubes with sulphuric acid (1 part of concentrated acid to 2 parts water), the resulting decomposition being titrated with permanganate, with the following results:—

	Per cent FeO.
0.5 grm. heated one day at 210° C...	.. 1'316
0.5 grm. heated two days at 230° C...	.. 1'416
0.5 grm. heated five days at 230° C...	.. 5'50

It seemed probable that this was not the total amount of ferrous iron in the columbite; hence attention was directed to an old method which is rarely used, yet seems to be worthy of greater attention than has been given it. Berzelius first suggested the method, though it is generally credited to Hermann. The finely ground mineral is mixed with fused and finely divided borax. A small platinum crucible is completely filled with this mixture, then covered with a platinum lid, and the whole placed in a larger platinum crucible. Dry magnesium oxide is packed around and over the inner crucible until it is completely covered, and so excluded from air contact. The heat of a good Bunsen lamp is applied for one-half hour, when the decomposition is complete. Longer heating, or too rapid cooling, causes the fusion to adhere very tightly to the crucible, and loss may result on endeavouring to remove it. When the whole is quite cold, the small crucible is taken out, freed from adhering magnesium oxide and weighed. The fusion, which is a clear green glass, is then freed from the crucible by sharply tapping; a piece may be broken off, weighed, ground in a mortar, dissolved in water and sulphuric acid, and titrated with potassium permanganate. Or, if the amount of ferrous iron is not large, it is better to crush the whole fusion in a diamond mortar, then place in a flask provided with a Bunsen valve, dissolve in water and sulphuric acid, and titrate. To prevent the oxidation of the iron during its solution, a quantity of sodium carbonate was placed in the flask with the ground fusion, and the water and sulphuric acid added carefully to this mixture. When a strong evolution of carbon dioxide had continued for several minutes, the cork carrying the Bunsen valve was quickly inserted, and the flask put aside until solution had taken place. It is necessary to shake the flask from time to time, otherwise the finely divided oxides which separate will enclose some particles of the fusion, and the result will be low. In one or two hours the insoluble residue should be a perfectly white, fine, homogeneous mass. The flask is then opened, more sulphuric acid added if necessary, and the iron titrated with permanganate. A number of fusions were made according to this method, the amount of ferrous oxide found being 6.426 per cent. The method seems to be, so far as columbite is concerned, perfectly trustworthy. It is rapid and the manipulation is not difficult. The oxides which separated out were perfectly white. In one experiment they were filtered off, washed with hot water, ignited, and weighed. The percentage of mixed oxides, 77.94 per cent, agrees quite well with that obtained by the bisulphate method.

The quantitative analysis of this columbite by fusing with bisulphate, as above described, gave the following results:—

	A.	B.	C.	D.	E.
Ta ₂ O ₅	..	78.61	79.04	79.00	77.96
Cb ₂ O ₅	..	78.61	79.04	79.00	77.96
TiO ₂	..	12.30	13.83	13.62	13.58
Fe ₂ O ₃	..	1.15	1.60	1.85	2.24
SnO ₂	..	8.96	8.32	—	8.08
WO ₃	..	101.02	102.79	—	100.86
MnO..	..	—	—	—	—

One-half grm. of material was used in each case. The ferric oxide, as given above, includes the ferrous, which, estimated by the method of Berzelius, equals 6.42 per cent.

In a sixth analysis 3 grms. of material were taken and due attention was paid to those constituents which former analyses had shown to be present, but in such small quantities that their determination was not trustworthy. The results in this case were:—

	Per cent.
Ta ₂ O ₅	 78.04
Cb ₂ O ₅	 78.04
TiO ₂	 0.24
WO ₃	 0.48
SnO ₂	 5.22
U ₃ O ₈	 6.42
Fe ₂ O ₃	 0.02
FeO ..	 8.96
CaO ..	 1.22
MnO..	
H ₂ O ..	
Total ..	 100.60

An interesting point in the composition of this columbite is the ferric oxide. Hermann records one analysis of some fragments of a columbite from Miask containing several per cent of it, and so far as I am aware this is the only columbite in which this constituent is mentioned. He also gives a Miask columbite containing 0.50 per cent of uranium oxide. Genth mentions a trace of uranium in a columbite analysed by him.

While no effort was made to separate the metallic oxides quantitatively, it was found from the preparation of pure material that the columbium was in decided excess. Titanic acid was proved to be present, and silica was found in very small quantities.

Many of the recorded analyses in which separations of columbic and tantallic oxides are given, fail to state whether any attempt had been made to eliminate or to prove the presence of titanium or silica. Given a mixture of tantalum, columbium, and titanium, the analyst will have no difficulty in separating tantalum from columbium by Marignac's double fluoride method. But the titanium double fluoride, when mixed with the columbium salt, shows an abnormal solubility which makes its separation very doubtful. This point will be more fully discussed later.

Fusion with Sodium Thiosulphate.—It occurred to me to try the decomposition of the mineral by fusion with sodium thiosulphate, believing that in this way tungsten and tin would be converted into sulpho-salts, and could then be more effectually removed from the other constituents. Without entering into detail, I may say the attempt was fruitless.

Decomposition by the Gibbs Method.—Some years ago Dr. Gibbs published a procedure (*Am. J. Sci. Arts*, xxxvii., 357, 1864) for the decomposition of the columbite minerals; and as my desire was to investigate the different methods of decomposition, I naturally turned to this suggestion. In mineral literature this course is given a second place to the bisulphate decomposition. My own

* From the author's thesis presented to the University of Pennsylvania for the degree of Ph.D., 1895. From the *Journ. Amer. Chem. Soc.*, xviii., January, 1896.

experience compels me to prefer it to the latter method. The details of the Gibbs method are, in brief, as follows :—

The mineral must be fine, yet need not be in an impalpable powder, as is necessary in the bisulphate decomposition; it was intimately mixed, by grinding in a mortar, with three times its weight of potassium fluoride; the mixture was transferred to a platinum crucible and made into a paste with concentrated hydrofluoric acid. The mass heated up at once, and for some minutes the decomposition proceeded without the application of heat. It was found advantageous to let this mixture of acid salt and mineral stand for several hours, stirring occasionally, and adding more acid if the mass became hard. It was then heated on a water-bath until the excess of acid was driven off. After thoroughly drying on an iron plate, the free flame was applied. Hydrofluoric acid was driven out of the acid potassium fluoride, and at length the whole mass fused and formed a clear, quiet, easily handled fusion, which, upon cooling, became a beautiful pink-violet in colour.

The decomposition is not complete until every part of the mixture has assumed this colour, which does not change on further heating. In the early part of the fusion a deep blue colour appears. If the action be interrupted at this point an incomplete decomposition will result.

The violet mass was taken up with water and hydrofluoric acid in a platinum dish, then boiled and filtered. This extraction should be repeated several times. If the decomposition is not quantitative, the solution in water is much hastened by first grinding the fusion. Any silica which may have been present in the mineral will remain as potassium silicofluoride. This being a gelatinous compound, it is likely to enclose fine particles of the fusion and prevent their solution. If the amount of silica is not large, a separation may usually be effected by treating with concentrated hydrofluoric acid; but if much silica be present it is safer to evaporate to dryness with a little sulphuric acid, and take up the remaining potassium sulphate with water. If any insoluble substance is left it may be dissolved in hydrofluoric acid and added to the main portion of the solution.

If an analysis of the mineral is desired, hydrogen sulphide gas may now be passed through the acid filtrate, whereby any tin, tungsten, or molybdenum present will be precipitated as sulphide. Filter, and separate as usual.

The filtrate was evaporated to dryness, and enough sulphuric acid added to expel all the hydrofluoric acid. The excess of acid was driven off on an iron plate, not over a free flame, and the oxides of columbium, tantalum, and titanium precipitated by boiling with a large quantity of water. The boiling must be continued for several hours to insure a complete precipitation, but it is not so difficult to bring down the metallic oxides under these conditions as in the bisulphate decomposition. Filter, and wash the oxides with hot water, first by decantation, then on the filter. The ignition of the oxides gave a perfectly white, fine powder; and this, fused with sodium carbonate or potassium fluoride, yielded a colourless mass when cold. The oxides obtained from the bisulphate never did so, but formed with the carbonate a tinge of green, and with the fluoride a tinge of pink, showing the presence of manganese, and probably of iron.

The filtrate from the mixed oxides contained iron, manganese, and uranium. These were separated by ammonium sulphide and ammonium carbonate, following the plan given under the bisulphate method.

When the object is simply the extraction of pure mixed oxides, the above procedure may be somewhat varied. The fusion is made just as usual, then taken up with water insufficient for perfect solution, and a small quantity of hydrofluoric acid, boiled, and filtered. On cooling, the filtrate will be found to be an almost solid mass of the columbium double fluoride, $2\text{KF} \cdot \text{CbOF}_3 \cdot \text{H}_2\text{O}$, which separates as a beautiful shining salt and consists of thin

laminae. At first the tantalum double fluoride remains undissolved, or is dissolved only in small quantity, as it is a very insoluble salt compared with the columbium compound, but if too much hydrofluoric acid is added the tantalum will be discovered with the columbium potassium fluoride, and larger amounts of iron and manganese will also contaminate it. From a very concentrated solution of the columbium double fluoride, such as would be obtained by this method, any tantalum double fluoride will, if present, separate almost immediately. These needles should be examined under a microscope for the thin transparent plates of the columbium salt. When these begin to appear filter at once and use a pump. The plates are a good indication that all tantalum is separated. The filtrate, on standing, will usually give the columbium salt, but it may have to be concentrated a little. The first crop of crystals may be coloured pink by manganese or iron. Re-crystallisation, however, removes this. The next crop is fairly pure. When working with large quantities a very satisfactory approximate separation of columbium from tantalum may be obtained by this method of extraction.

As boiling with pure water, or even with water containing a small amount of hydrofluoric acid, decomposes the tantalum potassium fluoride and leaves an insoluble compound, $2(2\text{KF} \cdot \text{TaF}_5) \cdot \text{Ta}_2\text{O}_5$, while the columbium double salt is practically unaffected, this treatment leaves us in the end a white, finely-divided mass which is almost free from columbium. By heating this residue on a water-bath with a rather concentrated solution of hydrofluoric acid and a little potassium fluoride, the tantalum potassium fluoride is obtained, and may be purified by re-crystallisation.

The Gibbs method was used for the preparation of rather large quantities of tantalum and columbium potassium fluorides. I think it preferable to the bisulphate decomposition and subsequent solution of the oxides in hydrofluoric acid in that it does not consume so much time, and iron and manganese are more readily eliminated. The only objection is that large platinum vessels are needed; as a substitute for these, rubber beakers and funnels were sometimes used.

The method finally adopted is as follows :—

Separation of Columbium and Tantalum by their Potassium Double Fluorides.—The pure mixed oxides were placed in a platinum crucible with three times their weight of potassium fluoride, then moistened with hydrofluoric acid as described under the decomposition of the mineral by the Gibbs method. By treating the fusion with water and hydrofluoric acid an almost perfect solution was obtained, since only a trace of silica was present. Concentration gave the long pointed needles of tantalum potassium fluoride, $2\text{KF} \cdot \text{TaF}_5$. These were filtered and the solution again concentrated. The crystal crop should be examined under the microscope, as it may be a mixture of tantalum and columbium. Usually it is only tantalum.

If a considerable excess of hydrofluoric acid and potassium fluoride is present in the mother-liquor, the next crop of crystals may be a complex mass about which the analyst can come to no definite conclusion. The fraction consists principally of long crystals much like the titanium double fluoride, and, to make the matter more puzzling, these crystals are not so soluble as those separating at the same time. They may be obtained pure by treating the mixture with a few drops of water and quickly filtering. Re-crystallisation from pure water gives the laminated salt $2\text{KF} \cdot \text{CbOF}_3 \cdot \text{H}_2\text{O}$. If the acid and potassium fluoride are not in large excess, usually two, and sometimes three, crops of the laminated salt are formed, but in time the long needles are almost sure to make their appearance. These needles were tested for titanium, but no satisfactory evidence of its presence was obtained.

When the solution is very concentrated large thin plates separate from it. These do not give the reaction

with gallotannic acid, but they react with zinc, hydrochloric acid, and potassium thiocyanate. This test for columbium compounds will be noticed later. Re-crystallisation does not give the laminated salt. The crystals are always found, and are by no means in small quantity. With zinc and hydrochloric acid they give a greenish colour which quickly becomes brown. They were repeatedly re-crystallised, then decomposed with sulphuric acid. The oxide obtained was white, and at 19° C. had a specific gravity of 4.57.

The oxide was placed in a platinum retort connected with a platinum condenser; hydrofluoric acid was poured over it, and a free flame was applied. The volatile products were collected in water in a platinum dish. Several evaporations were necessary for the volatilisation of this oxide. The solution in the dish was then treated with a small quantity of potassium fluoride and concentrated. The same large, thin plates crystallised out. These crystals were very beautiful, being frequently over an inch in length and 1½ inches in width. They were so transparent that often their presence in the dish was altogether unnoticed.

Analysis.

Substances taken. Grm.	K ₂ SO ₄ found. Grm.	Cb ₂ O ₅ found. Grm.
0.5000	0.5268	0.0059

This analysis would indicate that the salt is probably acid potassium fluoride with a small quantity of the double fluoride of columbium, yet it must not be forgotten that the reactions given above cannot be regarded as conclusive evidence of the presence of columbium.

Because of the brown colour with zinc and hydrochloric acid these crystals were also tested for titanium. Its presence could not be detected.

(To be continued).

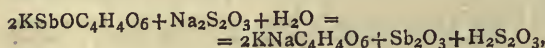
ON THE FORMATION OF ANTIMONY CINNABAR.

By J. H. LONG.

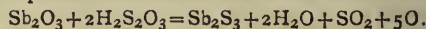
THE composition of the pigment known as antimony cinnabar has been stated by several different formulæ, as may be seen by consulting the leading hand-books of chemistry. The substance was usually considered as a mixture of sulphide and oxide or as an oxysulphide with the formula Sb₂S₂O. The formula Sb₂S₃ is found also in some of the older works, and Baubigny (*Comptes Rendus*, October 22, 1894) has shown that this is undoubtedly the correct one. Experiments made by myself, and described in this Journal in February, 1895, led me to adopt the same formula.

The compound is usually prepared by boiling a solution of antimony chloride or tartrate with sodium thiosulphate or crude calcium thiosulphate. As obtained from the acid solution of the chloride, the product is not pure and not of constant composition, being frequently mixed with oxychloride. This mixture is a mechanical one, and analysis made from it has no value in establishing a formula. The precipitate obtained by boiling a mixture of pure solutions of tartar emetic and sodium thiosulphate, on the other hand, has a constant composition, and numerous analyses I have made of it in the past year lead to the formula already given (*loc. cit.*).

By analogy with other formulæ established in the paper referred to, I suggested there that the reaction between the tartrate and thiosulphate may be represented by this equation:—



the oxide and thiosulphate then acting on each other to form sulphide:—



The oxygen and sulphur dioxide are not liberated as such, but held as polythionates with the excess of thiosulphate used.

To throw further light on the reaction I have attempted the formation of the cinnabar by other methods. While the product is sulphide of antimony, it appears that it can be made with its characteristic colour only by the decomposition of a thiosulphate. All attempts to obtain the true precipitate by action of hydrogen sulphide or alkali sulphides and sulphur dioxide on antimony solutions failed. The only body formed was the amorphous sulphide, often mixed with sulphur. On the other hand, by the action of a neutral or acid mixture of an antimony compound and a thiosulphate on each other, the cinnabar red product is the only one formed. If the mixture is made alkaline by the addition of a drop or two of ammonia water, no sulphide whatever precipitates. A small amount of hydrated oxide of antimony separates, but the decomposition of the thiosulphate is prevented. On the addition, now, of enough weak acid to neutralise the ammonia a yellow precipitate soon appears, but this speedily changes to deep bright red. The formation of the true cinnabar seems to begin by the appearance of a yellowish intermediate product, which is compatible with the above equations.

In this connection it is interesting to note the behaviour of pure antimony trioxide with solutions of thiosulphate. The reaction of the latter with a soluble antimony compound is comparatively rapid, and experiments were made to show the action of the oxide under the same conditions. It was found that the latter, when added to a strong or weak neutral thiosulphate solution, is unable to effect a decomposition in the cold or by application of heat. When the mixture is boiled the oxide remains perfectly white. This is true even after heating in an autoclave under a pressure of eighteen atmospheres.

It was found, however, that with the addition of a little acid to the mixture of oxide and thiosulphate a reaction followed after a time, although it never became complete. In a series of experiments a constant weight, 0.576 gm. of the pure precipitated, washed, and dried oxide was taken and mixed with water, and a constant weight of sodium thiosulphate in solution, in each case 0.992 gm. of the salt. Definite volumes of half normal hydrochloric acid were then added, and water enough to make the total volume 50 c.c. in each case. The mixtures were made in small Erlenmeyer flasks, loosely stoppered, and were very frequently shaken. The amounts of hydrochloric taken are given in the table below. The reactions became apparent only after several minutes, and, after five hours, had advanced so far in the mixtures numbered one and two, that the products had become orange. The reactions in the other flasks were less marked, but later became strong. The mixtures were made on October 7th, and were shaken many times daily through two months; in fact, as long as any change of colour in them was noticed. On December 3rd the amount of sulphide of antimony present was found by the method of Rivot, oxidation by chlorine after preliminary treatment with strong potassium hydroxide solution. The sulphur is found as sulphate, and the amount of sulphide formed in each case is shown by the table.

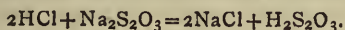
No.	Amount of Sb ₂ O ₃ . Grm.	Na ₂ S ₂ O ₃ . Grm.	N/2HCl. C.c.	H ₂ O. C.c.	BaSO ₄ found. Grm.	Sb ₂ O ₃ converted. Grm.
1	0.576	0.992	2	48	0.155	0.064
2	0.576	0.992	4	46	0.293	0.121
3	0.576	0.992	8	42	0.384	0.158
4	0.576	0.992	12	38	0.427	0.176
5	0.576	0.992	16	34	0.454	0.187

In mixtures one and two no evolution of sulphur dioxide could be detected by the odour or by tests, but in the

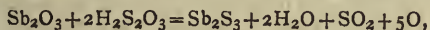
others it was apparent, weak in 3 and strong in 4 and 5. No free sulphur was precipitated in any case, or at any rate could not be found in the final product. Although but a small part of the oxide was actually converted, the colour of the products in mixtures 1 and 2 was a deep cinnabar, and perfectly characteristic. The amounts of sulphide formed or of oxide converted are not proportional to the volumes of acid used, and are much less than should be found on the assumption that the reaction begins by the production of antimony chloride from the oxide. If this were true, the sulphide formed by means of the soluble thiosulphate should increase with the acid taken. The reaction appears to take place between the oxide and thiosulphuric acid liberated by the hydrochloric acid, as was suggested by several experiments. In one case 0.500 grm. of antimony oxide was treated with 10 c.c. of half normal hydrochloric acid and 30 c.c. of water, as before, and allowed to stand twenty minutes, with frequent shaking. The mixture was then filtered, and to the filtrate 1 grm. of sodium thiosulphate in 10 c.c. of water was added. In a short time a precipitate of sulphur formed, perfectly light coloured, showing the absence of even a trace of the antimony. The rapidity with which the thiosulphate was decomposed showed that the hydrochloric acid taken must be in the filtrate and not in the residue, as oxychloride for instance. Titration of the filtrate showed this in a similar case. In a second experiment the acid and thiosulphate, in amounts equal to those of the last experiment, were mixed, and after the lapse of one minute the now opalescent mixture was added to some antimony oxide. Although the reaction between the first substances had gone into its second stage, showing that the hydrochloric acid was now certainly in combination, a precipitation of antimony sulphide began almost immediately, and in a short time the cinnabar colour was distinct.

Thiosulphuric acid is usually spoken of as quite unstable, but Landolt has shown (*Ber. d. Chem. Ges.*, xvii., 2958) that in dilute solutions it may exist many seconds, even minutes. The interval before precipitation is lengthened by dilution. If decomposition begins in presence of compounds of the heavy metals, a sulphide, sulphur dioxide, and polythionates may form. A large excess of thiosulphuric acid is necessary to complete the reaction in this manner, as suggested by the experiments of Vortmann (*Ber. d. Chem. Ges.*, xxii., 2307).

In Experiment No. 1 of the table above the amount of hydrochloric acid taken is just one-eighth of that necessary to complete this reaction with the thiosulphate:

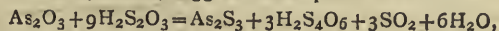


By full conversion enough acid would be liberated to complete the equation assumed at the beginning,



with the amounts of oxide and thiosulphate taken. It follows, therefore, that not over one-eighth of the antimony oxide taken should be found converted into sulphide, and the result of the experiment shows slightly less than this. According to this theory we should have 0.072 grm. of oxide changed. The test shows 0.064 grm. In the second and following experiments the amount of oxide converted is relatively still less. The acid taken in the last experiment is sufficient to decompose all of the thiosulphate and thus permit the conversion of all of the oxide. But the result shows that slightly less than one-third the oxide has been changed. In the first experiment no escape of sulphur dioxide was noticed, while in the last it was quite marked, and this fact has doubtless some connection with the low amount of sulphide formed. The reaction which takes place in a weak solution of thiosulphuric acid is evidently different from that in the strong solution, inasmuch as the greater portion of the sulphur seems to be given off as sulphide in the one case and as sulphur dioxide in the other.

In the somewhat similar reaction with arsenious oxide Vortmann (*loc. cit.*) suggests this equation—



in which but one-sixth of the sulphur present is used to form sulphide. By increasing the amount of hydrochloric acid added to the thiosulphate the decomposition of the latter is hastened.

It is possible that after a time, with increased liberation of sulphur dioxide, the formation of sulphide may be retarded, as was suggested by this experiment. I mixed half a grm. of the antimony oxide with one grm. of sodium thiosulphate in 10 c.c. of water, and added 10 c.c. of half normal hydrochloric acid and 30 c.c. of moderately strong solution of sulphur dioxide free from air. By using water instead of the last solution, precipitation would appear in a few minutes, but in this case it was delayed several hours, and then but a slight amount of yellowish product appeared. The thiosulphate is therefore protected from decomposition by the presence of the sulphur dioxide.

The cinnabar is easily formed from the oxychloride of antimony without addition of acid. Some recently precipitated and well washed oxychloride was mixed with water and thiosulphate solution of the strength used before. The characteristic colour soon appeared, and in a short time the whole product seemed to be cinnabar. The reaction is doubtless aided by the hydrochloric acid liberated by the decomposition of the oxychloride in presence of water. The acid in turn attacks the thiosulphate, and so the process becomes continuous and rapid. These reactions are all much hastened by application of heat and the quantitative relations are also altered, but at a temperature of 20° C. thiosulphuric acid seems to be the active precipitating agent in the cases investigated.—*Journal of the American Chemical Society*, xviii., No. 4.

DETECTION OF CAMEL IN WINES. POSSIBLE CONFUSION WITH THE COAL-TAR COLOURS.

By A. J. DA CRUZ MAGALHAES.

CAMEL is frequently employed—at least in Portugal—to give a facitious appearance of age to white liqueur wines. There exist methods for the recognition, and even for the quantitative determination, of caramel in wines, but we do not find in literature any indication as to the possible confusion between the coal-tar colours and those of caramel. Now this confusion may take place, and lead to grave errors.

In the course of some researches on the colouring matter of Portuguese wines I was led to try, on a liqueur wine from Oporto which, as I afterwards recognised, had been strongly caramelised, the general reactions given for the detection of coal-tar colours. The following are the results:—

1. I boiled 100 c.c. of wine for ten minutes with 10 c.c. of a solution of potassium sulphate and a flock of mordanted wool. The wool was dyed and retained its orange-yellow colour after plentiful washings in water and with ammonia.

2. 20 c.c. of wine were mixed with 10 c.c. lead subacetate and were filtered after agitation. The filtrate passed through with a distinct orange-red colour, and gave up its colour, on shaking, to amylic alcohol.

3. 100 c.c. of wine, supersaturated with ammonia, and shaken up with amylic alcohol, gave up their orange-yellow colour to this solvent.

4. 10 c.c. of wine were agitated (both in cold and in heat) with 0.2 grm. yellow mercury oxide for one minute, and filtered after settling. The filtrate was coloured an orange-yellow in each case.

We might therefore infer that the wine in question

was coloured an orange-yellow with one or more coal-tar derivatives.

I repeated the same experiments with a wine of the same type, of my own vintage, to which I had added pure caramel made with ordinary sugar. The results were absolutely the same.

On operating with the same wine without the addition of caramel nothing similar was obtained.

I repeated these experiments with pure caramel; the results obtained were exactly those to which I had added caramel. Without any doubt the colours of caramel may therefore be confounded with those derived from coal.

To throw light on a second point of this interesting subject, I prepared caramel from dextrose and saccharose, both very pure.

The two solutions of caramel thus prepared were treated with lead boric acetate and agitated with amylic alcohol. This remained colourless with dextrose caramel; with saccharose caramel the alcohol took a deep orange-yellow.

If both were supersaturated with ammonia and then shaken up with amylic alcohol, the first gave a greenish-yellow colour to the alcohol, and the second a very deep orange-yellow.

With the former ether takes no colour, but it takes an orange-yellow with the latter.

Mordanted wool took a yellow colour with the former, but an orange-yellow with the latter.

Cazeneuve's test does not alter the original colour of the two solutions.

The author is continuing the researches.—*Comptes Rendus*, cxxiii., No. 21.

THE OCCURRENCE OF TITANIUM.

By CHARLES E. WAIT.

It is not my present purpose to repeat what has been already frequently published relative to the presence of titanium in minerals, typical rocks, meteorites, clays, soils, blast furnace products, &c. I wish merely to call attention to the fact that some of the bodies with which we have much to do contain titanium, and that, probably owing to the difficulties formerly experienced in its estimation, it has been more frequently overlooked than is generally supposed.

In the recent examination of food materials, under the direction of the United States Department of Agriculture, I have had occasion to make analyses of the ashes of some plant materials, and, this having led to further investigations, I was interested and surprised to find titanium present in every plant ash thus far examined.

This is, in fact, surprising, as it is stated by some writers (Roscoe and Schorlemmer) "that it does not appear to form part of the animal or vegetable kingdom."

The amount of titanic oxide found in the ash of some vegetable material is as follows:—

Oak wood	0.31 per cent
Apple and pear wood (mixed) ..	0.21 "
Apple	0.11 "
Cow peas	0.01 "
Cottonseed meal	0.02 "

From the above determinations we are reasonably safe in assuming that titanium is assimilated by plants. If this is true, it seems very strange that reference to this fact has not been made by recent writers upon agricultural chemical analysis, and upon the chemistry of vegetable life.

In fact, in consulting treatises on ash analysis with tables (Wolff), I do not find any mention whatever of the presence of titanium. If this is a fact, can it be true

that it has escaped the attention of chemists for so long a time?

The examination of the ash of bituminous and anthracite coal shows the presence of titanic oxide. The results of some determinations are as follows:—

Jellico (Tenn.) bituminous coal	0.69 per cent
Coal Creek (Tenn.) bituminous coal ..	0.95 "
Pocahontas (Va.) bituminous coal	0.94 "
Middlesborough (Ky.) bituminous coal ..	0.83 "
Pennsylvania anthracite coal	2.59 "

With reference to the presence of titanic oxide in the ash of coal, it may be fairly assumed that, partly owing to the infiltration of clay and earthy materials, it would be found there, but is it fair to assume that its presence is wholly accounted for in that way? If mention has been made of the presence of titanium in the ash of coal, it has thus far escaped my attention.

The method employed in the above determination is that of A. Weller (*Ber. d. Chem. Ges.*, 1882), which is based upon the fact that hydrogen peroxide, when added to a solution of titanium, produces a compound of an intensely yellow colour. There are precautions necessary in the execution of this method which have already been pointed out (*Journ. Amer. Chem. Soc.* xiii., 210.)

It will be my pleasure to report additional notes at an early day concerning the presence of titanium in the vegetable kingdom. Valuable service has been rendered in the above work by Messrs. J. O. LaBach and C. O. Hill.—*Journal of the American Chemical Society*, xviii., p. 402.

GOVERNMENT LABORATORY OF TASMANIA.

By the courtesy of Mr. W. F. Ward, Assoc. Royal School of Mines, we have been favoured with the report of the Government Laboratory for the year 1895.

There has been a falling off in the number of samples analysed for municipalities and private individuals, though the work in the latter case is largely gratuitous, and in the former exclusively so. On the other hand the work done for the Government more than compensates this deficiency. The increase of duty on "oils" imported for eight months more than defrays twice over the total yearly cost of the laboratory. The departments of mines, railways, police, health, and agriculture obtain more or less analytical work free of cost. On the other hand, Customs' duties have to be paid even on the chemicals imported for the use of the laboratory. So that this department is much more than self-supporting.

The total number of samples analysed in the Government laboratories during the year 1895 was 2197, 1550 of which were teas, 12 of which were found adulterated or defective. Only one sample of coffee was examined and condemned as consisting one half of chicory. Seven samples of spirit of wine tested for the Customs were not sufficiently methylated, *i.e.*, not made undrinkable. On this we may remark that we have known methylic alcohol (not methylated spirits) be drunk to a serious extent by men engaged in dye and colour works. It does not appear that Tasmania has adopted the absurdity of requiring an addition of mineral "naphtha" to methylated spirits. The Tasmanian fiscal authorities, as well as those in the Home Kingdoms, should awake to the fact that a minimum of Dippel's animal oil renders alcohol undrinkable without interfering with its technical uses.

Nineteen samples of water have been analysed. The composition of two may, it is hoped, prove exceptional. One of them contained 0.98 part of albumenoid ammonia per million of water, as well as 1.3 grains per gallon of chlorine. Another contained 97 grs. per gallon of chlorine. Much of this was in the state of magnesium

chloride. The water was taken from the premises of a milk-vendor. Quackery flourishes in the Colonies as well as in the Home Kingdoms. A "self-cure" for nervous debility, advertised and sold at one guinea per ounce, was found to be merely Peruvian bark in powder, the present retail price per ounce being about one shilling!

A fatal case of poisoning with strychnine is cited. Thirty-six grs. of the poison were separated from the contents of the sugar basin. There is also mention of wholesale malicious poisoning in Queensland. There is no mention of poisoning with any substance unknown to European pharmacologists. In this direction much work remains to be done, and much of it might be done by local analysts.

It will not be deemed an unpardonable digression if we mention that malicious poisoning—as we learn from private sources—is exceedingly rife in South Africa. The deadly drug, or drugs, are introduced into the victim in the state of snuff, of which the Kaffirs are passionately fond. But they will now rarely accept a pinch from a stranger without careful scrutiny. To return to Mr. Ward's report, a number of samples of ores and tailings have been examined for the Secretary of Mines. In addition to gold and silver, nickel, cobalt, copper, tin, antimony, bismuth, zinc, and lead have been determined. We find no mention of platinum. A sample from M. Dundas contained silver at the rate of 1·057 ozs. per ton, and gold specimens gave 60 to 63 ozs. Arsenical pyrites are found, and prove a source of great annoyance.

PROCEEDINGS OF SOCIETIES.

ACADEMIE DES SCIENCES.

At the meeting of the Academie des Sciences, on Monday, Dec. 21st, M. A. CORNU delivered the following Presidential Address:—

When Lavoisier, Schwann, and Cagniard Latour studied the fermentation of beer, and Pasteur, taking up the question, patiently followed the development of those microscopic beings in generations called spontaneous in the diseases of wine or of silk-worms, who could have foreseen that a day would come when this admirable research would have so important a bearing on the welfare of humanity, demonstrating that these infinitely minute beings rank among the most important factors of human life. Pasteur, in his long and fruitful career, has taught us that it is possible to specify these organisms—to combat and even to direct them, according as they are our allies or our enemies. They have the power of conferring immunity or of conducting us infallibly to death.

The public see only final success; they mostly ignore the starting point of such researches; they ignore the efforts and the perseverance required to lead to what is vulgarly called a practical discovery; they are even prone to disdain abstract science, and to estimate the merit of the savant by the immediate market value of his discoveries.

Utilitarianism is, in fact, one of the maladies of the present age, perhaps one of the gravest, because it tends to crush the upward flight of the human spirit, and to fetter it to the exclusive worship of material interests.

Prof. Röntgen's discovery of the X rays has been, both for the public and for savants, the scientific event of the year. The speaker gave a luminous summary of the researches which have led to this discovery, laying due emphasis on the "brilliant experiments" of Mr. Crookes. The Röntgen rays are spoken of as a fresh benefit to be placed to the credit of pure science. After touching on the merit of the illustrious savants recently lost to the Academy and to science, the speaker mentioned that this

year the Arago medal has been conferred in duplicate on M. Antoine d'Abbadie and on Prof. Sir W. Thompson, now known as Lord Kelvin. He on a recent occasion had pronounced France as the *alma mater* of his scientific youth. It is added that modern nations, though bent under the yoke of material interests, and crushed under the treacherous law of iron and blood, are still able on great occasions to raise their eyes to the serene regions above hatred and envy and to join in celebrating the great men whose labours increase the common patrimony of intelligence at the same time as the well-being of mankind.

NOTICES OF BOOKS.

The Practical Methods of Organic Chemistry. By LUDWIG GATTERMAN, Ph.D., Extraordinary Professor in the University of Heidelberg, with numerous illustrations, translated by WILLIAM SHOBER, Ph.D., Instructor in Organic Chemistry in the Lehigh University. Authorised translation. New York: The Macmillan Company. London: Macmillan and Co., Ltd. 1896. Pp. 329.

WE have here an excellent work written in German, translated by an American, and printed in America. The language and the orthography employed are not in all cases idiomatic English as written and spoken on this side of the Atlantic. Thus we find magenta invariably designated by the term "fuchsine."

In the first or general part we have instructions for necessary operations, well thought out and clearly described. We may call particular attention to the directions for fractional distillation, to the management of autoclaves, and distillation at reduced pressures.

Next follow the methods for ultimate organic analysis, the determination of nitrogen being performed exclusively by the method of Dumas.

We now come to the special part; the performance of synthetical operations in the aliphatic, aromatic, pyridine, and quinoline series. Lastly comes an inorganic part, viz., instructions for obtaining in a state of purity substances required as reagents in organic research. It will be at once observed that the author does not give a series of recipes. He described representative reactions, e.g., the nitration of a hydro-carbon, the reduction of a nitro-compound to an amine, the reduction of a nitro-compound to an azoxy-azo or hydrazo compound. The student who has carefully worked out these directions in the laboratory will find himself prepared for entering upon the wide fields of organic research without losing his way. One who has made no such preparation is in danger of "messaging about" at random, and if he comes upon something valuable may not recognise what he has done.

We therefore strongly recommend the careful study of this work, regretting only that the distinguished publishers have not seen their way to having the English translation executed by an Englishman and brought out in England.

Catalogue of Chemical Apparatus, Balances, Drying-Ovens, Furnaces, Laboratory Stands, &c., also special and general Glass Apparatus, Hydrometers, Thermometers, Porcelain and Clay Ware, Jena Laboratory Glass Ware, and Glass Tubing. A. GALLENKAMP and Co., 2, 4, and 6, Cross Street, Finsbury, E.C. 1896.

AN important feature in this catalogue is the special description of Jena laboratory glass. This ware, we are told, surpasses the best Bohemian glass, Kavalier's make, in its insolubility in water, whether at ordinary temperature or at 80°. On treatment with caustic soda it is

slightly inferior to Bohemian glass, but it has the superiority as regards resistance to sodium carbonate. The Jena glass has a great power of resisting sudden changes of temperature. Medium sized flasks, containing boiling toluidin (200°), bear immersion in cold water. Jena laboratory glass can be heated over a Bunsen flame without wire gauze.

Evaporating basins are catalogued of Berlin, Thüringen, and Meissen porcelain, as well as of aluminium and enamelled steel, of pure nickel, platinum, silver, and platinum gold for melting potash. There is mention of autoclaves for bearing a pressure of 50 atmospheres, and apparatus for boiling in a vacuum or at a reduced pressure. Fletcher's devices for the production and distribution of heat are quoted in great variety. Apparatus for practical distillation as devised by Hempel, Linneman, Glinzky, and others, are also quoted and shown.

Of filter-pumps there is also good variety, of the constructions of Fischer, Bunsen, Volhard, Finkener, Geissler, Alvergriart, and others, as also filtering apparatus in conjunction with pumps.

Various kinds of filter paper are quoted, some of which retain such precipitates as zinc sulphide.

The balances and weights described are those of Sartorius, Becker, Verbeck and Peckholdt, and Beranger.

Bacteriological apparatus is figured and described at great length.

Spectroscopes figure here at length unusual in catalogues of laboratory requisites. Catalogues of microscopes, including the renowned instruments of Zeiss, will, it is said, be supplied on application.

Of chemicals we find only a list of standard solution. Experimentalists may often find here some newly devised apparatus which will prove exceedingly useful. We cannot help expressing our regret that Messrs. Gallenkamp find themselves unable to get their printing done in Britain.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Revue Universelle des Mines et de la Metallurgie.
Series 3, Vol. xxxv., No. 2.

Uses of Acetylene.—R. K. Duncan proposes to set out from acetylene to form, either by way of polymerisation of benzene, C_6H_6 , whence there may be obtained the innumerable series of aromatic compounds, or ethylene by the action of nascent H. Acetylene, on account of its endothermic character and its explosive properties, cannot compete with coal gas or electricity for the lighting of cities. This compound was first met with by Davy in 1836, and described in full by Borchsenius in 1891. It yields per kilo. 280—300 litres of acetylene gas. Acetylene is less poisonous than ordinary coal gas. Its liability to explosion is a serious obstacle to its use on the large scale.

Action of Coal Gas upon Caoutchouc.—Gossheintz.—Black tubing is the least suitable, then the red quality, but the grey kind is the best.

Treatment of Rich Iron Ores and Use of Acid Slags.—Franz Büttgesbach.—The author has made use of ores containing 68 to 70 per cent of iron, with the addition of sufficient slags free from iron to such an extent that the slag may reach at least 25 per cent of the cast metal.

Series 3, Vol. xxxv., No. 3.

Copper Industry in Japan.—Japan now occupies the fourth rank among the copper-yielding countries. In 1892 it produced 20,000 tons of copper. The ore is distributed over the entire country. The yield will soon exceed that of Chili.

Assistant Wanted in a London Laboratory.—Full particulars to Box M., CHEMICAL NEWS Office, Boy Court, Ludgate Hill, London, E.C.

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THE CHEMICAL NEWS.

Vol. LXXV., No. 1938.

A RAPID DETERMINATION OF THE EQUIVALENT OF SULPHURIC ACID AND ITS PLACE IN THE TEACHING OF CHEMISTRY.

By WILLIAM ACKROYD, F.I.O.

THE quantitative experiments on neutralisation usually given to elementary students are few in number, and of one type; the following variation is suggested as part of a scheme of quantitative work.

a c.c. of aqueous sulphuric acid are found to be neutralised by *b* c.c. of solution of sodium hydroxide.

To *a* c.c. of the acid a weighed quantity of magnesium is added (from 0.1 to 0.2 gm.), and when all action has ceased *b* c.c. of the alkali are added. The indicator used (methyl orange) shows that all the free acid has been neutralised, and it may readily be proved that all the metal is precipitated as hydroxide. Now run in, from the burette, sulphuric acid of known strength until the magnesium hydroxide is exactly neutralised.

From the data obtained calculate the equivalent of sulphuric acid.

Example.—Strength of sulphuric acid used, 0.04945 gm. per c.c. 20 c.c. of the acid requires 21.5 c.c. of alkali for neutralisation. 0.146 gm. of magnesium is dissolved in 20 c.c. of the acid, and after addition of the necessary 21.5 c.c. of alkali 12.4 c.c. of the acid are required to neutralise the magnesium hydroxide. Whence, taking 12 to be the magnesium equivalent, we get—

$$\frac{12.4 \times 0.04945 \times 12}{0.146} = 50.4.$$

Other students obtained the numbers 49.9, 49.7, 48.7 &c., and the figures give some idea of the degree of accuracy to be expected under ordinary circumstances. The time taken for the estimation is much under an hour.

By neutralising the magnesium hydroxide with hydrochloric acid of known strength, the equivalent of this acid may be similarly ascertained. Results within a unit of the accepted number are readily obtained.

A recent alteration of the syllabus of the Science and Art Department has introduced the salutary course of taking quantitative work from the commencement instead of leaving it until the student's third year. These first efforts are necessarily only, in many cases, rough approximations, but they serve to familiarise the worker with the laws of combination in definite and reciprocal proportions, equivalents, &c. The methods to be employed, and their order, are left to the discretion of the teacher, who has the help of such excellent little works as those of Ramsay, Reynolds, and Tilden.

I take it that in an ideal course for the elementary student each quantitative experiment ought to be preceded by the qualitative work necessary for its comprehension, and it ought to require as little time as possible for its performance, so that it can be tried often; the experiments ought to follow one another in logical order, each one more or less built on those going before, with the continuity found in a series of geometrical problems, and collateral work in theory and practice should proceed abreast with them.

The determination of equivalents will form the backbone of the work, and magnesium, because of its purity and rapidity of action, lends itself admirably for the purpose, as in the following experiments:—

1. Determination of the magnesium equivalent by finding what weight of it is required to liberate 11.16 litres of hydrogen at normal temperature and pressure, *i.e.*, 1 gm. from sulphuric acid.

2. Determination of the oxygen equivalent by finding how much of it combines with 12 grms. of magnesium in the course of ignition of the latter in a porcelain crucible.

3. Determination of the sulphuric and hydrochloric acid equivalents by finding what weight of each is required to neutralise the hydroxide formed from 12 parts by weight of magnesium.

4. Determination of the equivalent of caustic soda by finding what weight of it neutralises an equivalent of sulphuric acid.

In this order, where each experiment is based on a preceding one, the equivalents 1 (assumed), 12, 8, 49, 36.5, and 40, would be obtained for H, Mg, O, H₂SO₄, HCl, and NaOH respectively, and each experiment is typical of others which will suggest themselves to the teacher. I think it is of importance to stick to the unit weight of hydrogen for comparisons, as subsequently there is nothing to unlearn. Where many units of other elements are employed in empirical comparisons, as, *e.g.*, the ratio 1 : 0.66 in experiment 2, instead of 12 : 8, much confusion arises in many minds, and the ratio 12 : 8 is equally available as an example of combination in fixity of proportions.

It appears therefore inadvisable to follow the historic order in this respect with elementary students, however interesting it may be when they have a better grasp of the subject.

OPTICAL ANALYSIS OF URINE AND EXACT DETERMINATION OF THE PROTEIDS, THE GLUCOSIDES, AND THE NON-FERMENTIBLE SACCHAROID MATTERS.

By F. LANDOLPH.

1. *Sugar in Urine*.—Normal healthy urine always contains from 0.01 to 0.20 gm. sugar per litre; the exact determination can only be effected by fermentation. Setting out from 0.40 gm. of sugar per litre, the physician should turn his attention to the slow and progressive development of diabetes, which may be considered established when 2 grms. per litre of fermentible sugar is found. There are only pathological urines containing albumen, pus, &c., which often contain no trace of sugar.

2. *Optical Determination of Sugar*.—The saccharimeter is generally incompetent to demonstrate the presence of 1 gm. to 2 grms. sugar per litre, because normal urine always deflects from 1° to 3° to the left. It is only on setting out from 2° to 3° deviation to the right that we are almost certain of the presence of sugar in the urine, and it is only beyond 10 grms. per litre that the diabetometer gives us fairly exact results; the more exact the higher the quantity. Hence, to obtain results beyond dispute, it is indispensable to have recourse to fermentation, whilst for quantities above 20 grms. per litre the two methods give approximately the same results.

3. *Direct Coefficient and Indirect Coefficients of Reduction*.—The direct coefficient of reduction can only be obtained with urine boiled and filtered, because raw urine in treatment with the cupopotassic liquid always holds a certain quantity of cuprous oxide in solution. To 10 c.c. of urine boiled and filtered we use 10 c.c. water and 40 c.c. of Fehling's solution. We heat the mixture to ebullition, and keep it up, when once the reaction has commenced (which generally requires from three to five minutes), for twenty minutes; we filter, wash the cuprous oxide with boiling water, dry, and ignite. The weight of copper oxide obtained, calculated per thousand, gives

the direct coefficient of reduction; the third of this weight gives fairly well the quantity of non-fermentible saccharoid matter in a litre of urine, deducting the quantity of oxide corresponding to the fermentible sugar and the quantity of oxide corresponding to the uric acid, of which one part is approximately equal to four parts of copper oxide.

A quantity of non-fermentible saccharoid matter exceeding 3 grms. per litre is the certain sign forerunning diabetes. Further, for these salts of urine with strongly diabetic disposition the duration of the introduction of the reaction often does not exceed half a minute.

To obtain the indirect coefficients of reduction, we first split up (at first in raw urine, and then in a portion boiled and filtered), the mucine and analogous proteids, as well as the glucosides of mineral acids. We then fix in the urine thus treated, filtered, and adjusted to their primitive volume, the coefficients of reduction as for the determination of the direct coefficient of reduction. The difference between the figures of the two indirect coefficients gives the quantity of mucine in copper oxide, and the difference between the direct coefficient of the boiled and filtered urine and the indirect coefficient of the same urine boiled and filtered gives the quantity of the glucosides in copper oxide, the third part of which represents the weight of these compounds.

4. *Polaristrobometric Examination of Urine.*—When a urine contains pus and analogous pathogenic elements, the deflection to the left in the very sensitive polaristrometer of Pfister and Streit becomes stronger, reaching 5° , and even 8° , which is evidently due to the polarising force of the nuclei of the granulated leucocytes of pus. In this case it even happens that the field of vision becomes totally obscure in an extent of some degrees. This fact is especially very important when the cellulæ and pus granules have already disappeared under the microscope, since this procedure alone permits us to know if there has been an anterior presence of pathogenic elements or not.

Analogue studies are pursued to recognise and determine the nitrogenous organic compounds preceding albuminuria.—*Comptes Rendus*, cxxiii., No. 26.

THE SEPARATION OF VANADIUM FROM ARSENIC.

By CHARLES FIELD and EDGAR F. SMITH.

As vanadium and arsenic occur associated in minerals and likewise in artificial products, their separation becomes a matter of consequence.

The course usually pursued in carrying out this separation is that long since recommended for the removal of vanadic acid from its solutions; namely, its precipitation as ammonium metavanadate. Other methods have recently appeared in the literature bearing on analysis. Reference is here made especially to the publication of Fischer ("Bestimmung von Vanadinsäure," Dissertation, Rostock, 1894).

Experiments made in this laboratory on the behaviour of vanadates (*Journ. Am. Chem. Soc.*, xvi., 578) and arsenates (*Ibid.*, xvii., 682) heated in an atmosphere of hydrochloric acid gas, in which both acids were volatilised, suggested the thought that if the sulphides of vanadium and arsenic were exposed to the same vapours perhaps they would show a variation in deportment. And so it has proved. Perfectly dry arsenic trisulphide, previously washed with alcohol, carbon disulphide, and ether, then dried at 100°C ., when exposed in a porcelain boat, placed in a combustion tube, was almost completely expelled from the retaining vessel at the ordinary temperature. The last traces were driven out at a temperature little above 150°C . Brown vanadium sulphide, in a perfectly dry condition, treated in the same manner, was not

altered. It only remained then to prepare mixtures of known amounts of the two sulphides and subject them to the action of the acid vapour. To this end the following experiments were made:—

- I.—0.1303 grm. of vanadium sulphide,
0.1302 grm. of arsenic sulphide.

The arsenic sulphide was volatilised without difficulty and left 0.1297 grm. of vanadium sulphide.

- II.—0.1290 grm. of vanadium sulphide,
0.2242 grm. of arsenic sulphide,

gave after exposure of one hour to hydrochloric acid vapour a residue of vanadium sulphide, weighing 0.1297 grm.

- III.—0.0828 grm. of vanadium sulphide,
0.0582 grm. of arsenic sulphide,
left 0.0827 grm. of vanadium sulphide.

- IV.—0.1306 grm. of vanadium sulphide,
0.2028 grm. of arsenic sulphide,
gave a residue of 0.1308 grm. of vanadium sulphide.

- V.—0.1403 grm. of vanadium sulphide,
0.2409 grm. of arsenic sulphide,
left 0.1404 grm. of vanadium sulphide.

The temperature in these experiments was not allowed to exceed 250°C ., as beyond that point there is danger of affecting the vanadium and causing its partial volatilisation.

The method worked so well and with such evidently favourable results that the following course was adopted in the analysis of a specimen of the mineral vanadinite. 0.2500 grm. of air-dried and finely divided material was placed in a porcelain boat; the latter was then introduced into a combustion tube and gently heated in a current of dry hydrochloric acid gas. By this treatment vanadic and arsenic oxides were expelled, leaving lead phosphate and chloride. The receiver containing the vanadium and arsenic was made alkaline and digested with ammonium sulphide. From the solution of the sulpho-salts the vanadium and arsenic sulphides were set free by a dilute acid. After washing and careful drying these sulphides were separated as indicated in the preceding lines, then changed to oxides and determined in the usual manner. The sum of the total constituents determined as lead oxide, phosphoric oxide, vanadic and arsenic oxides, with some lead chloride, amounted to 0.2501 grm.

The method, in addition to being satisfactory in the analytical way, certainly forms a very excellent means of purifying and freeing vanadium from arsenic.—*Journal of the American Chemical Society*, xviii., No. 12.

THE SEPARATION OF MANGANESE FROM TUNGSTIC ACID.

By WALTER T. TAGGART and EDGAR F. SMITH.

THE necessity of obtaining pure tungstic acid from time to time, using wolframite as the starting out material, has frequently suggested the inquiry as to what course would probably prove the best in the quantitative separation of this acid from oxides, such as those of iron and manganese.

In the experiments recorded in this communication only the results obtained from a study of mixtures of a mangano-salt and a soluble alkali tungstate will be given. The directions taken in the experimentation were, 1st, to effect the separation by the use of yellow ammonium sulphide in the presence of ammonium chloride; 2nd, to eliminate the acid oxide by the use of an alkaline carbonate.

Following the first course, mixtures of definite amounts of ammonium tungstate and manganous chloride were made. To these was added water and a considerable

excess of yellow ammonium sulphide, together with ammonium chloride. The mixtures were digested on a water-bath at 70° C. for several hours, and the vessels containing them were then closed and allowed to stand during the night. The manganese sulphide was filtered out, and, after solution, was changed into sulphate and weighed as such, or it was finally obtained as protosesquioxide in the customary way.

Results.

Manganous oxide present.	Manganous oxide found.
Grm.	Grm.
0.1950	0.2121
0.1949	0.2255
0.1290	0.1708
0.1287	0.1720
0.1291	0.1760

In every trial tungstic acid adhered to the metallic oxide.

In trying the second suggestion the soluble tungstate and the soluble manganous salt were digested for some hours in a platinum dish, upon a water-bath, with an excess of a 10 per cent potassium carbonate solution, after which the whole was evaporated to dryness, the residue boiled up with water, the manganous carbonate filtered out, washed, and finally converted into protosesquioxide.

Results

Manganous oxide present.	Manganous oxide found.
Grm.	Grm.
0.1949	0.1516
0.1949	0.1534

Several trials were made using a 50 per cent solution of potassium carbonate.

Results.

Manganous oxide present.	Manganous oxide found.
Grm.	Grm.
0.1951	0.1745
0.1950	0.1528

The experimental evidence given in the preceding paragraphs leaves no doubt as to the insufficiency of the two methods which were tried in effecting the desired separation. It is probable that fusion with an alkaline carbonate will alone answer for this purpose. How complete that course would be can only be ascertained by careful experimentation.

In the course of analysis molybdenum is quite often obtained as sulphide. Its conversion into a weighable form is attended with more or less difficulty. Trials made in connection with its estimation show that if the sulphide, as generally obtained, be dried, then intimately mixed with anhydrous oxalic acid, its careful ignition to trioxide can be made quite rapidly.

Results.

Molybdenum trioxide taken.	Molybdenum trioxide found.
Grm.	Grm.
0.3000	0.3009
0.3000	0.2990
0.1007	0.1011

—*Journal of the American Chemical Society*, xviii., No. 12.

On Dibromo 1-3 Propene. — R. Lospieau. — Epibromhydrine, β : $\text{CHBr}=\text{CH}-\text{CH}_2\text{Br}$, is a colourless liquid which irritates the eyes and the skin. Its density at 0° is 2.097. If cooled to -75° it congeals, but afterwards melts at -52°. It boils at 155-156°. Its molecular weight is = 200. — *Comptes Rendus*, cxxiii., No. 25.

THE SEPARATION OF BISMUTH FROM LEAD.

By ARTHUR L. BENKERT and EDGAR F. SMITH.

MANY methods have been suggested to effect this separation. In a recent issue of the *Zeitschrift für Angewandte Chemie* (1895, p. 530), Olav Steen reviews thirteen of these methods, and concludes that an early proposal of Rose (*Ann. Chem. Phys. Pogg.*, cx., 425), in which the lead is thrown out as chloride and weighed as sulphate, another by Löwe (*Journ. Prakt. Chem.*, lxxiv., 348), in which the bismuth is removed as basic nitrate, and a late suggestion made by Jannasch (*Ber. Chem. Ges.*, xxv., 124), viz., the expulsion of the bismuth as bromide from a mixture of lead and bismuth sulphides by an air current carrying bromine, are the most satisfactory. At least these methods gave Steen the best results. The separation of bismuth from lead frequently confronts the analyst, and any novelty in this direction cannot be absolutely devoid of interest; hence the present communication, which brings data that may perhaps prove of service in the hands of others who are interested in the solution of this analytical problem.

It will be recalled that Herzog (*Ztschr. Anal. Chem.*, xxvii., 650) proposed to separate bismuth from lead by precipitating the former as basic acetate. The method required considerable time for execution, and in other hands than those of its author apparently has not yielded entirely satisfactory results.

An idea closely related to that of Herzog would be the substitution of a formate solution for that of the acetate. This was done, with results that are very interesting.

Solutions of lead nitrate and bismuth nitrate in nitric acid were made up of such strength that 20 c.c. of the first contained 0.2076 gm. of lead oxide, and 20 c.c. of the second 0.1800 gm. of bismuth trioxide. The lead and bismuth were accurately determined after dilution to a litre. 20 c.c. of these two nitrate solutions were then introduced into a beaker glass, carefully diluted and almost neutralised with sodium carbonate, or until the incipient precipitate dissolved slowly, when considerable sodium formate solution of sp. gr. 1.084 and a few drops of aqueous formic acid were added. The total dilution of the liquid was 250 c.c. It was gradually heated to boiling and held at that point for five minutes. The precipitate was then allowed to subside, but was filtered while yet hot. The basic formate separates rapidly, and is easily washed if not boiled too long. It was washed with hot water, then dissolved in dilute nitric acid, and precipitated with ammonium carbonate. The ignited bismuth trioxide weighed too much; it contained lead. However, the impure oxide was dissolved in nitric acid, diluted to 250 c.c., and after the addition of sodium carbonate to almost complete neutralisation, sodium formate and free formic acid were added as before, and the precipitation of basic formate repeated. This precipitate after solution and the bismuth thrown out by ammonium carbonate, gave 0.1804 gm. of bismuth oxide instead of 0.1800 gm. as required by theory. Seven additional separations, in which the quantities of bismuth and lead were the same as indicated above, gave—

0.1806 gm. of Bi_2O_3 .
0.1806 " "
0.1803 " "
0.1804 " "
0.1804 " "
0.1805 " "
0.1796 " "

The conditions in these determinations were similar to those previously outlined.

With a solution containing 0.3600 gm. of bismuth oxide and 0.2076 gm. of lead oxide, operating in an analogous manner, two results were obtained:—

0.3595 gm. of Bi_2O_3 .
0.3605 " "

instead of the required 0.3600 gm.

The residual bismuth trioxide was examined for lead, but none was found.—*Journal of the American Chemical Society*, xviii., No. 12.

DETERMINATION OF THE ATOMIC MASSES OF SILVER, MERCURY, AND CADMIUM, BY THE ELECTROLYTIC METHOD.*

By WILLETT LEPLEY HARDIN.

Introduction.

A GLANCE at the literature on the determinations of the atomic masses of silver, cadmium, and mercury, will show that, with the exception of cadmium, the electrolytic method has not been tried. Aside from the fact that certain errors involved in the washing and drying of the precipitates are eliminated by this method, its simplicity at once gives it preference over the usual methods of gravimetric determinations. Inasmuch as these three metals are completely precipitated from certain of their solutions by the electric current, and as it is desirable to determine the atomic mass of any element by different methods, it was thought advisable to apply this method in a re-determination of the atomic masses of these elements.

General Considerations.

Before taking up the different metals separately, the following general considerations may be mentioned:—

1. A careful preliminary study was made in the selection of compounds. Some compounds, which from a theoretical standpoint seemed to offer certain advantages, were found by experiment not to meet the requirements of exact determinations. Salts which can be sublimed were used whenever possible; and in all cases only those salts were used which form well-defined crystals.

2. All reagents used were either prepared or purified by myself, and carefully tested for impurities.

3. The metals were deposited in platinum dishes of about 200 c.c. capacity and about 65 grms. in weight. When the precipitation was complete, before interrupting the current, the solution was syphoned from the platinum dish, pure water being added at the same time: this was continued until the solvent used was completely removed from the dish. The current was then interrupted, and the deposit washed several times with boiling water, with the hope of removing any occluded hydrogen. After drying, the dishes were placed in a vacuum desiccator over anhydrous calcium chloride, and allowed to remain in the balance room until their temperature was the same as that of the room. Atmospheric dust was excluded from the platinum dishes during the process of deposition by means of two glass plates which formed a complete cover; the moisture which collected on this cover was washed back into the dish from time to time. The dishes were handled with nickelled tongs tipped with rubber.

4. The balance used was made expressly for this work by Henry Troemner, of Philadelphia. The beam and pans were made of aluminum, the beam being about 20 c.m. long. The framework was plated with gold to prevent corrosion. The sensibility for different loads and the ratio of the length of the two arms were carefully determined. The balance is sensitive to the fortieth of a m.grm., and the sensibility is almost independent of the load up to 75 grms. The difference in the length of the two arms is so slight that no correction need be applied. The balance was kept in a large quiet room of nearly constant temperature.

The larger weights used were made of brass, and the fractions of a grm. made of platinum. The weights were

all previously compared against each other, and standardised with reference to the largest weight. The small corrections found in comparing them were tabulated and applied to all results. The weighings were made by the method of oscillations. The temperature and barometric pressure were noted at the time of each weighing, and all weighings were reduced to a vacuum standard. As the density of the atmosphere at the time of weighing the empty platinum dish was different from that at the time of weighing the dish and deposit together, the following formula was applied to obtain the weight of the deposit *in vacuo*:—

Weight of (dish + deposit) —

$$\frac{\text{weight of dish} \left(1 + \frac{\lambda}{\Delta} - \frac{\lambda'}{f} \right)}{1 + \frac{\lambda'}{\Delta} - \frac{\lambda'}{f}} \times \left[1 + \frac{\lambda'}{\Delta} - \frac{\lambda'}{f} \right] = \text{weight of deposit in vacuo.}$$

Where λ = density of air at the time of weighing the empty dish.

λ' = density of air at the time of weighing the dish + deposit.

Δ = density of platinum dish.

Δ' = density of metallic deposit.

f = density of weights.

As the weights were all standardised with reference to the 100-grm. brass weight, it is evident they must all be calculated as having the same density, equal to that of brass.

5. The atomic masses of the different elements involved in the calculation of results were taken from Clarke's latest report (*J. Am. Chem. Soc.*, xviii., 197).

PART I.

Determination of the Atomic Mass of Silver.

The mean of all the earlier determinations, as calculated by Clarke, gives 107.923 for the atomic mass of silver,—a result almost identical with the mean (107.93; O = 16) of the determinations of Stas.

Preparation of Pure Metallic Silver.

The silver used in this work was purified by the Stas method. Two hundred grms. of silver, about 99 per cent, pure, were dissolved in dilute hot nitric acid. The solution was evaporated to dryness, the nitrate heated to fusion and maintained in a fused condition until the oxides of nitrogen were no longer evolved. The residue, after cooling, was dissolved in as little cold water as possible, and after standing forty-eight hours the solution was filtered through a double filter to remove any suspended matter. The clear solution was then diluted with thirty times its volume of distilled water, and to it was added an excess of pure hydrochloric acid. The silver chloride which separated was allowed to subside, and was then thoroughly washed by decantation, at first with water containing a little hydrochloric acid, and finally with pure water. The precipitate was then collected on a cheesecloth filter, pressed strongly, and allowed to dry. When perfectly dry, the silver chloride was powdered finely and digested for three days with aqua regia; it was then thoroughly washed by decantation with distilled water. After obtaining the pure chloride of silver, it was necessary to purify the caustic potash and milk-sugar used in reducing the chloride to the metallic state. The caustic potash was heated to the boiling-point, and to it was added a concentrated solution of potassium sulphide to precipitate any heavy metals which might be present. The

* Contribution from the John Harrison Laboratory of Chemistry No. 13. From the author's thesis presented to the Faculty of the University of Pennsylvania for the degree of Ph.D.—From the *Journal of the American Chemical Society*, xviii., p. 990.

solution was filtered, and the filtrate digested for some time with freshly precipitated silver oxide, and again filtered to remove the excess of potassium sulphide. The milk-sugar was purified in a similar manner. The silver chloride was then placed in large porcelain dishes and covered with a solution of caustic potash and milk-sugar. The dishes were placed on a water-bath, and heated to a temperature of 70° to 80° until the reduction to finely divided metallic silver was complete. The alkaline solution was then poured off, and the grey metallic silver was washed with distilled water until the alkaline reaction disappeared. The metal was then digested with pure dilute sulphuric acid, and finally washed with dilute ammonia water. The silver thus obtained was mixed, when dry, with 5 per cent of its weight of fused borax containing 10 per cent of pure sodium nitrate. The mixture was fused in a clay crucible and the silver poured into a mould. The metal obtained in this way was almost snow-white in appearance, and dissolved completely in nitric acid to a colourless solution.

Preparation of Pure Nitric Acid.

To obtain pure nitric acid, one-half litre of the commercial C. P. acid was mixed with an equal volume of concentrated C. P. sulphuric acid and distilled from a retort provided with a knee-tube and condenser. The first portion of the distillate was rejected. The process was stopped when half of the nitric acid present had been distilled over. The distillate was mixed with an equal volume of pure sulphuric acid and re-distilled. The second distillate was collected in a flask, the mouth of which was closed with glass wool. When the process was complete the flask was closed with a doubly perforated cork, and placed in a water-bath at a temperature of 40° . A current of pure dry air was then conducted through the acid to remove any oxides of nitrogen. The acid was kept in a dark place.

Experiments on Silver Oxide.

If pure, dry silver oxide could be prepared, the atomic mass of silver could be compared directly with that of oxygen. A large number of experiments were made on this compound with the hope of determining the ratio of the atomic masses of these two elements.

Preparation of Silver Oxide.

A portion of the pure metallic silver was dissolved in pure dilute nitric acid, and the solution evaporated to crystallisation. The crystals of silver nitrate were dissolved in pure water, and to the solution was added a solution of pure sodium hydroxide, prepared by throwing pieces of metallic sodium on distilled water in a platinum dish. The 25 grms. of silver oxide prepared in this way were washed by decantation with 20 litres of water. The material was then dried at the ordinary temperature, after which it was finely powdered and dried for twenty-four hours in an air-bath at 100° . The oxide was kept in a weighing tube in a dark place.

Several analyses were made by dissolving a weighed portion of the material in pure potassium cyanide, electrolyzing the solution, and weighing the resulting metallic silver. The observations invariably gave less than 95 for the atomic mass of silver. The oxide was re-dried at a temperature of 125° , and analysed as before, but the quantity of silver obtained was far below that calculated for the compound Ag_2O . Observations were also made on material dried at 140° and 150° . The results showed that it was impossible to prepare the silver oxide in a pure, dry condition.

After making these observations my attention was called to an article by Carey Lea (*Am. J. Sci.*, xlv., 240), in which were given the results of a series of analyses of silver oxide dried at different temperatures varying from 100° to 170° . These observations prove conclusively that oxygen is given off at a much lower temperature than that required to remove the last traces of moisture. From these observations and the results obtained by my-

self, it was evident that any further attempt to determine the atomic mass of silver from the oxide would be useless.

Although no careful study was made as to the nature of this compound, it might be added that, from my own observations, it seems very probable that the oxide contains some hydrogen in the form of hydroxyl.

(To be continued).

THE MANUFACTURE OF CALCIUM CARBIDE.*

By J. T. MOREHEAD and G. de CHALMOT.

(Continued from p. 18).

TAKING into account the weight of the product, the time in which it has been produced, and the number of horse-power used, we calculate for each run the amount of pounds produced per horse-power in twenty-four hours. By multiplying these figures by the number of cubic feet of gas produced per pound we obtained the number of cubic feet of gas produced per horse-power in twenty-four hours. In the accompanying table we give the results of experiments, wherein everything has been determined, wherein both unslacked and slacked lime have been used, and voltage, ampère, and duration of runs were varied. Since these results were obtained we have had many visitors from all parts of the country, and for each party we have made a test run. The results of these runs have all confirmed our previous results, with one exception, which was due to the presence of $5\frac{1}{2}$ per cent magnesia in the lime.

It is obvious that the results obtained from unslacked lime are far better than those with air-slacked lime. This is undoubtedly due to a loss of power used in decomposing the hydrated lime. The unslacked lime used by us contained, after being ground, from 5 to 9 per cent of water. In practice it is necessary to use the mixture that comes from the furnace again. This mixture always contains some carbonate of lime; but if it be mixed when still hot with the necessary amount of carbon, and put again into the furnace, the lime has no opportunity to slack. The unslacked lime has the further advantage that it weighs less and is much less bulky, and that the mixtures made from it cool much faster than those made from slacked lime. The only disadvantages of unslacked lime are, that it must be ground, and that mixtures made from it require more stoking if put into the furnace. The mixtures of unslacked lime can stand up against the sides of the furnace under a very steep incline, and they can leave a hole all around the pencils. The mixture to be used should, on an average, contain 100 parts of lime and 64 to 65 parts of carbon, in order to obtain a carbide of about 5 cubic feet of gas per pound. If the voltage is increased to 100 it is better to take a little more carbon (100 lime and 66 to 67 carbon). If the voltage is 65 or less, 63 to 64 parts of carbon are sufficient. If the amount of carbon is increased the carbide becomes purer, but there is often more coating.

The largest amount of gas per horse-power is obtained if the carbide yields about 5 cubic feet of gas per pound. The yield of carbide in pounds varies inversely with the quality. In the following table we give the results of a series of experiments made with slacked lime and with a current of 65 volts and from 1700 to 2000 ampères. Several of these experiments have not been taken up in Table II., because the amount of slag on all the pieces of carbide has not been determined.

Carbide has been made successfully in Spray by the use of both the direct and the alternating current. We cannot express an opinion as to what current can be used

* Read Sept. 3rd before the Springfield meeting of the A.A.A.S. by one of us (M.). We have made since then several additions, so as to make the article complete up to the present time. From the *Journal of the American Chemical Society*, April, 1896.

TABLE II.—Unslacked Lime.

Date.	Time of experiment. Hours.	Volts.	Ampères.	Loss of voltage in the pencils. Per cent.	Horse-power.	Production in 24 hours, including slag. Pounds.	Net production. Pounds.	Cubic feet of gas per hour.	Cubic feet of gas per horse-power in 24 hrs.
June 27	2'50	100	1700	7'0	214	9'87	9'42	4'83	45'50
July 2	3'00	100	1666	8'0	205	10'34	9'76	5'25	51'24
" 1	2'25	100	1700	10'0	205	10'66	10'10	4'66	47'06
June 24	3'20	100	1600	7'0	214	11'50	10'73	4'93	52'90
" 28	2'50	100	1700	10'0	205	11'70	11'10	4'75	52'72
July 18	3'00	65	2000	5'0	165	9'63	9'15	4'95	45'29
" 19	3'00	65	1900	5'0	158	10'40	9'62	4'83	46'46
" 5	3'75	65	2000	5'0	165	8'38	8'15	5'40	44'01
" 9	4'50	65	2000	5'0	165	9'50	9'05	4'99	45'16
Aug. 10	6'00	65	1800	8'0	144	9'34	9'00	5'39	48'51
" 13	6'00	65	1800	8'0	144	10'83	10'44	4'82	50'32
July 31	7'00	75	1800	8'5	166	11'44	10'53	4'83	50'86
Average ..								9'75	48'33
								4'97	48'33

TABLE III.—Air-slacked Lime.

					Per horse-power.		Per pound.	
June 25	5'00	100	1700	7'0	214	8'34	7'96	5'30
" 29	4'00	100	1700	10'0	205	8'78	8'34	4'98
" 19	5'50	100	1700	7'0	214	9'25	—	4'89
" 22	4'00	100	1600	7'0	199	9'80	9'65	4'74
Aug. 14	4'50	75	1700	7'0	159	7'88	7'13	5'50
July 12	3'75	85	1800	3'5	198	—	6'33	5'55
" 26	8'00	75	1800	5'0	172	8'40	7'23	5'33
" 12	5'50	85	1775	8'0	185	—	7'32	5'32
" 6	5'00	80	1020	3'0	200	8'78	8'16	5'11
" 23	3'00	65	1800	5'0	150	6'83	6'40	5'78
" 22	2'00	65	1800	5'0	150	7'13	6'40	5'62
" 22	2'00	65	1800	5'0	150	7'20	6'40	5'64
" 25	9'00	65	1800	5'0	150	7'72	7'27	5'54
" 20	4'00	65	1800	5'0	150	8'60	8'00	5'01
" 23	5'00	65	1800	5'0	150	9'02	8'00	5'07
" 24	8'00	65	1800	5'0	150	9'30	8'03	4'97
Average ..								7'51
								5'27

TABLE IV.

Date.	Production per horse-power including slag. Pounds.	Cubic feet of gas per pound.
July 23	6'85	5'78
June 14	7'10	5'80
July 22	7'13	5'62
" 22	7'20	5'64
" 25	7'72	5'54
Aug. 14	7'88	5'50
May 21	8'10	5'20
" 22	8'30	5'10
July 26	8'40	5'33
June 4	8'46	5'52
July 20	8'60	5'01
June 5	8'76	4'94
May 28	8'80	5'20
" 23	8'82	5'10
July 23	9'02	5'07
June 8	9'06	5'10
" 24	9'30	4'97
July 11	9'30	4'33
Aug. 12	9'44	4'51
May 31	9'87	4'30
Aug. 8	10'52	4'23

to the best advantage, for we are not able to compare results. All of the results communicated in this paper have been obtained by the use of the alternating current. That electrolysis plays a part in the carbide manufacturing process of Mr. Wilson is therefore out of the question, and we do not need to use a furnace of the Moissan construction to prove this. It is not desirable to increase the ampère over 2000 if only six carbons of 4 inches square are used. The higher the ampère the greater the loss of voltage in the pencils, and therewith that of power. The carbons will also last longer if the ampère is low, because they do not become so hot. Lastly, we did not

obtain as great a yield per horse-power if the ampère was high and the voltage correspondingly low. We obtained the best yield of gas per horse-power by using a current of 100 volts, which can be seen by comparing the average of the results given in Table II.

TABLE V.

	Volts.	Horse-power.	No. of experiment.	Average cubic feet of gas per horse-power in 24 hours.
Unslacked lime ..	100	205—214	5	49'88
	65—75	144—165	7	47'23
Slacked lime ..	100	200—214	3	43'15
	75—85	159—100	5	38'71
	65	150	7	38'55

It must be taken into account that we measured the primary current, and that the losses of ampère in the transformers probably have been higher when we did not use the highest voltage, *i. e.*, 100. We do not know in how far it would be advisable to increase the voltage over 100, since our dynamos cannot give us a current of more than 100 volts. We believe, however, that the heat yielded by an arc of 100 volts, and from 1700 to 2000 ampères, is about the largest amount to be profitably used for the production of carbide in one furnace with six pencils, as it is used in Spray. We base our assumption on the following facts:—The quality of the carbide becomes better if the voltage decreases. We experienced some trouble in obtaining large carbide crystals with an arc of 100 volts and 1700 ampères, and in order to obtain a carbide that yields more than 5 cubic feet of gas per pound the mixture should contain an excess of carbon. If a current of 100 volts and 1700 ampères is used, the furnace requires more attendance and stoking than if a lower power, and especially a lower voltage, is used. The higher the voltage the faster the pencils must be

raised, for if the voltage is low (50 or 65) the carbide spreads out much more than if the voltage is high (100 volts). In the latter case the carbide builds up as a long thin piece, and it is oftener necessary to empty the furnace. As to the time that one furnace should be used continuously, we wish to say that we did not perceive a difference in the quantity and quality of the product whether we ran three hours or from three to nine hours. We must, however, remark that in the case where we used 100 volts and 1700 ampères with mixtures of unslacked lime we could not continue running for much more than three hours, because the construction of the furnace did not admit of raising the pencils quite 3 feet. With slacked lime we made also with this high power very satisfactory runs of five and five and a half hours. During the first hour the production is somewhat lower. It seems that more heat is lost probably for heating up the furnace.

The mixture used in all of the following experiments contained lime 50·08 per cent, and coke (of 92·17 per cent of carbon), 39·22 per cent. The current was of 65 volts and 1800 ampères, the loss of voltage in the pencils 5 per cent, and the net horse-power 150.

TABLE VI.

Time of experiment. Hours.	Production per hour in pounds.	Cubic feet of gas per pound.	Cubic feet of gas per hour.
1	37	5·63	208·31
2	40	5·62	224·80
2	40	5·64	225·60
3	40	5·78	237·20

(To be continued).

DERIVATIVES OF COLUMBIUM AND TANTALUM.*

By MARY ENGLE PENNINGTON.

(Continued from p. 20).

Qualitative Reactions.

THROUGHOUT this investigation the following questions constantly arose: How shall the purity of the columbium and tantalum compounds be determined. When is columbium free from tantalum? When is it free from titanium?

In the earlier work upon columbium we find Hermann describing a new element which he obtained from the mother liquors of the columbium potassium fluoride. This element, he states, gave a dark brown solution when reduced with zinc and hydrochloric acid, while the pure columbium compound gave a blue colour. Both these solutions, on standing in the air, reverted to the white hydrate. Marignac replied that the brown colour was not due to ilmenium, but to titanium, a view which is now generally accepted.

He also declares that a brown colour is produced when the potassium columbium oxyfluoride is treated with zinc and hydrochloric acid, the acid being in considerable excess. Then, by titrating with permanganate, he found that an intermediate oxide had been formed, to which he gave the formula Cb_2O_5 .

Crystals of the columbium salt, prepared as described above, continued to give this brown solution even after they had been subjected to five or six re-crystallisations. Following the plan of Krüss and Nilson (*Ber. d. Chem. Ges.*, xx., 1676), the atomic value of the oxide contained in such crystals was determined by decomposing with sulphuric acid, weighing the pentoxide and the potassium sulphate, then, by the ratio $2K_2SO_4 : Cb_2O_5$, determining

the value for Cb_2O_5 . This was found to be 85·7. Iron and manganese has been eliminated; titanium therefore was the probable cause of this low atomic value. The salt used was perfectly white, yielding a pure white oxide. This oxide was tested for titanium by the most delicate reactions known for the metal, but its presence could not be proved by any of them.

1. *Colour and Reduction Reactions.*—It has been found that the qualitative tests given in the various text-books for these three elements do not always hold good when the solution used is a double fluoride. As it is in this form that the separations are usually made, it has been thought advisable to note the action of some of the common reagents on these salts.

Gallotannic acid, which is considered the most characteristic test for columbium salts, behaves differently with different double fluorides. An acid solution of the laminated salt gives almost instantly a deep brick-red precipitate. The salt, crystallising in long needles, gives a lighter red precipitate which does not separate so rapidly. The large, thin, transparent plates previously mentioned give only a slight precipitate, and this is yellow in colour. These reactions are most delicate if the salt be dissolved in water, a drop of hydrochloric acid added, then a little gallotannic acid dissolved in alcohol. After standing several hours all the precipitates assume the same colour—a dark brick-red.

Tantalum double fluoride gives a sulphur-yellow colour with gallotannic acid. This, however, on standing, becomes brick-red, as the columbium does.

Titanium compounds are said to give a brownish colour with gallotannic acid, which changes quickly to an orange-red. The potassium titanium fluoride gave a straw-yellow colour with this reagent; in time a flaky precipitate forms, but the colour does not materially alter.

The following colour reaction serves for the detection of very small quantities of columbium, and is applicable to any soluble columbium compound. An excess of potassium thiocyanate is added to a small quantity of the dissolved substance; then some pieces of zinc followed by strong hydrochloric acid. At once the solution becomes a bright golden brown, which, if much columbium be present, may be almost red. A brisk and continued evolution of the gas does not alter this tint, which is also stable for more than twenty-four hours in the acid solution. Neither titanium nor tantalum give any reaction with potassium thiocyanate under the above conditions.

Hyposulphurous acid, H_2SO_2 , gives noteworthy colour reactions with these salts. The tests were conducted in the following manner:—A few cubic centimetres of a concentrated solution of sulphur dioxide were placed in a test-tube provided with a cork, and granulated zinc was added. The liquid changed to a greenish colour, and hydrogen was liberated. As soon as the evolution of the gas had ceased the solution containing the hyposulphurous acid was poured into the salt solution to be tested.

A solution of titanium double fluoride gave an orange-yellow colour at once. The oxide, when treated in like manner, became yellow.

Columbium double fluoride gave no colour, but a white hydrate was soon precipitated. Columbic oxide gave a slight yellow tinge.

Tantalum double fluoride gave no colour, but after standing a white precipitate separated. Tantalic oxide remained colourless when treated with hyposulphurous acid.

The white precipitates from the tantalum and columbium salts were probably hydrates due to the oxidation of the acid and its consequent action upon these salts.

Zinc and hydrochloric acid gave no reaction with the double fluoride of tantalum. With titanium a clear delicate green was obtained. The columbium salts always gave a colour with these reagents. The solution is at first dark blue, then a greenish brown, and finally a dark

* From the author's thesis presented to the University of Pennsylvania for the degree of Ph.D., 1895. From the *Journ. Amer. Chem. Soc.*, xviii., January, 1896.

	2KF.CbOF ₃ .H ₂ O.	2KF.TaF ₅ .	2KF.TiF ₃ .
Lead acetate.	White precipitate.	White precipitate.	White precipitate.
Mercuric chloride.	Slight precipitate in 24 hours.		
Mercurous nitrate.	Yellow precipitate.	Yellowish-green precipitate.	Yellowish-green precipitate.
Potassium chromate.	White precipitate, soluble in H ₂ O. Partly soluble in K ₂ CrO ₄ solution.		Precipitate soluble in water.
Potassium bichromate.		Precipitate after standing.	
Potassium cyanide.	White precipitate on boiling.	White precipitate.	White precipitate.
Potassium ferrocyanide.	Green-blue precipitate on boiling.	Yellow precipitate on boiling.	Precipitate on boiling.
Potassium thiocyanate.	White precipitate.	White precipitate soluble in the cold. Comes down by boiling.	
Potassium iodide.	White, granular precipitate. Iodine is liberated.	White granular precipitate.	No precipitate, but iodide is liberated.
Disodium hydrogen phosphate.		White precipitate after standing.	White precipitate.
Silver nitrate.		White precipitate after standing.	
Sodium bisulphite.	White precipitate.	White precipitate.	White precipitate.
Sodium pyrophosphate.		Slight cloudiness.	Precipitate.
Hypophosphorous acid.			Precipitate.
Sodium metaphosphate.			
Potassium bromide.	White precipitate.	Slight cloudiness.	

brown. Frequently a brown precipitate separates, which, on standing, becomes white.

The hydrochloric acid solution of columbic oxide, and also the potassium columbium fluoride, were tested with hydrogen peroxide, this being accepted as one of the most delicate reagents for titanium. No yellow colour in either case was obtained.

2. *Reactions with Wet Reagents.*—A number of the ordinary reagents have been tried with these salts, the results being given in the table above. The reactions for the greater number are very different when the metal tested is as double fluoride. The ferrocyanides, in particular, have quite abandoned their ordinary colours with these compounds.

Disodium hydrogen phosphate, when added to titanium double fluoride, precipitates the titanium completely. The filtrate, tested with ammonium hydroxide, gave no precipitate. Columbium double fluoride, on the contrary, is not affected by this reagent. After boiling a long time in a platinum dish a few white flocks were observed in the solution, but in such small quantity that they were disregarded. Whether this behaviour may or may not be made the basis of a separation of these two elements is not yet determined, because of the difficulty in getting rid of the phosphoric acid. Fusion with sodium carbonate, extraction with water, and subsequent precipitation by sulphuric acid gives a mixture of sodium salt and columbic oxide. Some columbium remains in solution. Fusion with potassium acid sulphate is more satisfactory, yet is not complete.

Deposition of Tantalum, Columbium, and Titanium Double Fluorides toward the Electric Current.

1. A solution of potassium columbium double fluoride, 2KF.CbOF₃.H₂O, in water, was treated with a small amount of sodium acetate. The precipitate formed was dissolved in acetic acid, and through this solution a current of one ampère, obtained from a thermopile, was conducted for five hours. A white precipitate, seemingly a hydrate, was formed. On breaking the current, this rapidly went into solution.

2. (a) A solution of the salt in water was subjected to the same current for eight hours. Almost immediately the bottom of the platinum dish was covered with a blue deposit. This gradually spread over the whole surface exposed to the action of the current, and became in a short time iridescent. As the deposit increased, the deep blue tint changed to more of a grey,

and remained so until the current was broken. It was washed quickly with water, then with alcohol, and it was dried on the hand.

0.1315 grm. of the salt was taken; the deposit weighed 0.0282 grm. This metallic-looking substance did not alter in the air, but, on subjecting it to a red heat, a white, shining, apparently crystalline compound resulted. It was readily soluble in hydrofluoric acid.

(b) A second experiment, with 0.2195 grm. of the substance, gave, under the same conditions, a deposit weighing 0.0388 grm. This, when ignited in the air, burned to a white oxide weighing 0.0312 grm. The blue compound is, in all probability, a lower hydrated oxide of columbium.

3. The electrolysis of an aqueous solution of a sodium columbate gave a white flocculent hydrate, not adherent to the dish. The precipitation was not complete. A current of one ampère was employed for a period of seven hours.

4. With a much stronger current (two ampères), a solution of the double salt 2KF.CbOF₃.H₂O, gave first a white hydrate, then, beneath the outer edge of the anode appeared a dark brown ring which gradually grew in towards the centre of the dish, never reaching it, however, but stopping when about half an inch in width.

This brown substance was slightly adherent to the dish, but just as soon as the current was broken, and the liquid poured off, it reverted to the white hydrate. This change was so rapid that it was impossible to separate the brown from the white substance.

Thinking that this brown compound might be a contaminating element, about 1 grm. of the double salt was dissolved in water and electrolysed until the brown ring had appeared. Then the liquid was poured into another dish as quickly as possible, and the current run through again. The brown ring appeared as before, and was treated in the same manner. After changing the dish four times only a trace of brown could be seen. When the remaining solution was evaporated it was found that almost the entire quantity of the columbium had been precipitated. The brown substance here formed resembles in its behaviour that produced in a like solution by zinc and hydrochloric acid.

The resistance of this solution is very high.

5. Potassium tantalum fluoride, in aqueous solution, was subjected to the action of a current of two ampères for six hours. A small quantity of hydrate was found in

the liquid, and on the dish a very slight iridescent deposit mixed with some white hydrate.

6. Potassium titanium fluoride was treated in the same manner as the previous salt. A small quantity of hydrate was found here, some of which adhered to the dish. The iridescent deposit, however, was wanting.

Action of Hydrofluoric Acid upon the Oxides of Tantalum, Columbium, Titanium, and Silicon.

The well-known volatility of the oxides of tantalum and columbium, when heated with hydrofluoric acid, led to the hope that in this behaviour might lie a separation from titanium, and also from silica.

Rose states that a very appreciable loss occurs when the first two oxides are treated as suggested, but he makes no attempt to separate them from the latter two. To this end 1 grm. of the mixed oxides of tantalum and columbium was evaporated to dryness with hydrofluoric acid, the residue being heated over the free flame for a few minutes. By this treatment dense white vapours were driven off. Upon weighing the residual oxides they were found to equal 0.5464 grm. A second evaporation gave further loss, but as both columbium and tantalum continued to remain, the method is without value.

The separation of silica from these oxides can be accomplished by the heat of an iron plate after evaporating to dryness on a water-bath. The final heating must be carefully done, and the acid should not be in too great excess.

I have never found it impossible to dissolve either the mixed or the pure oxide in hydrofluoric acid, even though strongly ignited. It is true, concentrated acid is necessary, and a little time is often required, but a perfect solution does take place.

Tantalum oxide, containing columbic oxide, is far more soluble in hydrofluoric acid than the pure oxide. The same behaviour has been observed with pure columbic oxide, though it is not so pronounced as with tantalum oxide. Titanium dioxide, ignited, is very difficultly soluble in this reagent, though columbic oxide, containing titanic oxide, went quickly into solution.

Double Fluorides of Tantalum, Columbium, and Titanium, with Rubidium and Cesium.

The potassium double fluorides of tantalum and columbium have been found of great service in separating these two metals. Marignac first showed that a separation could be effected through these salts, and he also demonstrated that the sodium and ammonium salts were inapplicable.

Of the potassium double fluorides of tantalum and columbium we possess considerable information. A number have been isolated and studied. The sodium salts crystallise so poorly that their history is not so well known. It seemed probable that rubidium and cesium would form double fluorides of definite crystalline character with these three metals. At least, a study of their behaviour might be found instructive. Before taking up their preparation, however, the simple fluorides of rubidium and cesium may be discussed.

Rubidium Fluoride (RbF).—An examination of the literature on rubidium showed that its fluoride had not been prepared. In order to procure this rubidium iodide was dissolved in water, and moist silver oxide added to precipitate the iodine. The solution of rubidium hydrate resulting was filtered off and evaporated in porcelain dishes. A very appreciable quantity of silver oxide was held in solution by the rubidium hydroxide, so that it was necessary to evaporate it almost to dryness, then to take it up in the smallest possible quantity of water, and filter. This treatment may have to be repeated two or three times before the solution is perfectly colourless. When quite free from silver, the concentrated solution was made slightly acid with hydrofluoric acid, and evaporated. If hydrofluoric acid be present it is almost impossible to obtain crystals, a thick syrup being formed which defies

all attempts in this direction. The solution is therefore evaporated with water several times until the excess of acid is expelled. The rubidium fluoride then crystallises in long, transparent plates. These were drained, and dried between filter paper. The salt was anhydrous. Conversion into sulphate by evaporating with sulphuric acid gave, from 0.5 grm. of the salt, 0.5236 grm. rubidium sulphate. This corresponds, therefore, to the formula RbF.

Cesium Fluoride.—Cesium chloride was dissolved in water, and the chlorine precipitated by moist silver oxide. The solubility of the oxide of silver in cesium hydrate is even greater than in rubidium hydrate, therefore some difficulty was experienced in obtaining a hydrate free from silver. It was finally accomplished by evaporating to dryness repeatedly, taking up the cesium hydrate in a very small quantity of water, and filtering it. The pure hydrate was then neutralised with hydrofluoric acid and evaporated. A thick syrup was obtained which refused to crystallise. Upon heating in an air bath to 130° C., a crystalline mass formed, but it was always in such a sticky condition, and absorbed moisture so rapidly, that it could not be analysed satisfactorily. This mass was dissolved in water and added to the solutions of the metals in hydrofluoric acid.

Double Fluoride of Columbium and Rubidium.—One-half grm. of columbic oxide was dissolved in hydrofluoric acid and the calculated quantity of rubidium fluoride added. The solution was evaporated on a water-bath to expel the excess of acid. The residue was taken up in hot water and allowed to crystallise spontaneously. White microscopic plates separated. These were filtered off, dried between filter paper, and analysed. Two-tenths grm. of the dry salt gave—

	Found.	Calculated for 2RbF.CbF ₃ .	Difference.
Cb ₂ O ₅ ..	0.0670	0.0673	-0.0003
RbF ..	0.1048	0.1049	-0.0001

The formula of the salt is therefore 2RbF.CbF₃, corresponding to the tantalum salt usually obtained with potassium fluoride.

The filtrate from this first crop of crystals was slightly concentrated, when small, shining, or even iridescent crystals, apparently plates, separated. Upon standing a short time, these changed over into crystals like those first mentioned. This salt is very soluble in water containing hydrofluoric acid, and also in pure water. It is insoluble in alcohol.

Double Fluoride of Rubidium and Tantalum.—Rubidium fluoride in slight excess was added to tantalum oxide dissolved in hydrofluoric acid. Small white needles crystallised out. An excess of acid must be present, otherwise heat decomposes the double salt, giving a fine, white, insoluble compound, as is the case with the potassium salt.

Analysis of $\frac{1}{10}$ grm. gave

	Found.	Calculated for 2RbF.TaF ₅ .	Difference.
Ta ₂ O ₅ ..	0.0915	0.0913	+0.0002
RbF ..	0.0861	0.0859	+0.0002

Double Fluoride of Titanium and Rubidium.—The preparation of this salt was conducted as described with preceding salts. The crystals here were also microscopic needles. Some difficulty was at first experienced in completely drying the salt, but this was overcome by several re-crystallisations from pure water, when an anhydrous product was obtained. One-tenth grm. of the salt gave, on analysis—

	Found. Grm.	Calculated for 2RbF.TiF ₄ . Grm.	Difference.
TiO ₂ ..	0.0238	0.0240	-0.0002
RbF ..	0.0622	0.0626	-0.0004

Double Fluorides of Tantalum and Cæsium.—This double salt was formed by the addition of a solution of the cæsium hydrate in hydrofluoric acid to a solution of tantalum oxide in hydrofluoric acid. Very beautiful white needles separated, which were not easily soluble in water, and were not decomposed by re-crystallisation from pure water. The aqueous solution may be evaporated on a water-bath with perfect safety, this salt being apparently much more stable than either the potassium or rubidium salt.

The crystals were dried in the air, then heated to 125°C . in an air-bath. No loss in weight was observed. 0.25 grm. gave, on analysis—

	Found.	Calculated for $15\text{CsF} \cdot \text{TaF}_5$.	Difference
Ta_2O_5 ..	0.0212	0.0217	-0.0005
CsF ..	0.2232	0.2228	-0.0004

The formula deduced from the analytical data varies widely from that generally followed by tantalum double fluorides. Neither is it in accordance with Remsen's law for the double halides (*Amer. Chem. J.*, ii., 291), though it will be observed that its fluorine content bears a simple ratio to the fluorine in combination with the tantalum.

Double Fluoride of Columbium and Cæsium.—This double salt was formed in the manner described for the preparation of the cæsium tantalum fluoride. It is very soluble in water containing hydrofluoric acid, and in pure water, from which it crystallises in needles. These, when pure, are anhydrous. Boiling with pure water does not decompose the salt. Analysis of $\frac{1}{10}$ grm. gave the following result:—

	Found.	Calculated for $7\text{CsF} \cdot \text{CbF}_3$.	Difference.
Cb_2O_5 ..	0.0216	0.0213	+0.0003
CsF ..	0.1694	0.1693	-0.0004

This salt, which appears to be $7\text{CsF} \cdot \text{CbF}_3$, is even more erratic in its constitution than the tantalum cæsium compound. There is apparently no relation here between the fluorine in combination with the columbium and the number of molecules of cæsium fluoride present.

Double Fluoride of Titanium and Cæsium.—This salt separates in very small, shining crystals when cæsium fluoride is added to a rather concentrated solution of titanic oxide in hydrofluoric acid. It is more readily soluble in water than the tantalum cæsium compound, and is not decomposed by pure water. The air-dried crystals showed no loss in weight after heating for some time at 125°C . An analysis of 0.25 grm. gave the following amounts of titanic oxide and cæsium fluoride:—

	Found.	Calculated for $4\text{CsF} \cdot \text{TiF}_4$.	Difference.
TiO_2 ..	0.0269	0.0271	+0.0002
CsF ..	0.2071	0.2076	-0.0005

The figures point to the formula $4\text{CsF} \cdot \text{TiF}_4$. This is a departure from the usual titanium double fluorides, and agrees with the law laid down by Remsen for these salts.

When we consider the atomic masses of tantalum, columbium, and titanium, the first 182, the second 94, and the third 48, and also consider the quantities of cæsium fluoride which unite with a molecule of each of the metallic fluorides, we find that with tantalum the quantity (fifteen) is nearly twice that with columbium (seven), and the latter almost double that (four) uniting with titanium, just as 182 is about twice 94, and 94 nearly twice 48.

These new cæsium compounds tend to confirm the conclusion drawn by Wells and others (*Amer. Journ. Sci.*, xlvii.) from their work on the cæsium double halides. The compounds investigated by these chemists show that the cæsium double halides are not wholly conformable to Remsen's law.

* This and all the other cæsium double fluorides are being subjected, at this writing, to further study in this laboratory.—E. F. SMITH.

The method of analysis pursued for the determination of these double salts is, briefly, as follows:—

The dry substance was decomposed in a platinum crucible by a few drops of concentrated sulphuric acid. The hydrofluoric acid was driven off, and the excess of sulphuric acid was then expelled on a sand-bath. The temperature must be just sufficient to drive off the acid. The metallic oxide was obtained from the sulphate by long boiling with a large quantity of water. It was then filtered, washed about twenty times with boiling water, ignited, and weighed. The filtrate containing the alkaline sulphate was evaporated, the excess of acid neutralised with ammonium carbonate, and the solution then evaporated to dryness on a water-bath. A saturated solution of ammonium carbonate was added, and the mixture evaporated again to dryness. The ammonium salts were expelled by careful heating. Constant weight can generally be obtained after two or three evaporations with ammonium carbonate. The rubidium sulphate decrepitates on heating, which necessitated great care while expelling ammonium salts, and also rendered the method proposed by Krüss (heating in a stream of ammonia gas) untrustworthy. The alkalis were then weighed as normal sulphate, and the cæsium or rubidium content calculated. This method, while slow, has been found very satisfactory for these rare alkalis.

(To be continued).

OBITUARY.

THE LATE DR. EMIL DU BOIS REYMOND.

THIS most distinguished physiologist concluded his fruitful and honourable career on December the 26th. The deceased, perhaps our highest authority in the wide and interesting region of animal electricity, was born at Berlin on November 7th, 1818. As his name indicates, he was of French descent. He studied firstly theology, at the University of Berlin, but soon turned his attention to the more congenial subject of medicine, becoming one of the most prominent pupils of Johannes Müller, and soon acquired—as, indeed, he richly merited—a world-wide celebrity. In 1851 he was elected a member of the Royal Prussian Academy of Sciences, and in 1867 he was appointed Perpetual Secretary, an office which he filled up to his death. His eminence was needed as a counterpoise to the quackery which was and is still seeking to degrade the study of animal electricity.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiii., No. 26, December 28, 1896.

An interesting ceremony took place at the Institute Pasteur on the transfer of the remains of the late illustrious *savant* to the crypt specially designed for his reception. The principal personages present were:—M. Rambaud, the Minister of Public Instruction, on behalf of the Government; M. Baudin, President of the Municipal Council, on behalf of the city of Paris; Sir Joseph Lister, President of the Royal Society of London, Foreign Associate of the Academy of Sciences, on behalf of the Royal Society, and of the Royal College of Surgeons of London; Sir W. Priestley, on behalf of the Universities of Edin-

burgh and St. Andrews; Sir Dyce Duckworth, on behalf of the Royal College of Physicians of London; M. A. Cornu, President of the Academy of Sciences; M. Bergeron, Perpetual Secretary of the Academy of Medicine; M. L. Passy, Perpetual Secretary of the Society of Agriculture; and M. Duclaux, Director of the Institute Pasteur.

New Application of Radioscopy to the Diagnosis of Maladies of the Thorax.—Ch. Bouchard.—Details of some cases of no chemical interest.

The Energy expended by a Muscle in Static Contraction for the Maintenance of a Charge after Respiratory Exchanges.—A. Chauvureau, with the collaboration of M. Tissot.—The conclusion from the authors' first series of experiments is, that the quantities of oxygen absorbed and of carbonic acid exhaled—*i. e.*, of the energy expended for the maintenance of a charge—increase with the muscular contraction, although the charge remains constant. The conclusion from the second series of experiments is, that the oxygen absorbed and the carbonic acid exhaled—*i. e.*, the energy brought into play for the maintenance of a charge—increases sensibly in the same manner as such a charge.

New Radioscopic Facts concerning Intrathoracic Lesions.—J. Bergonié.—This paper has no chemical bearings.

Action of Lithium upon Carbon and certain Carbides.—M. Güntz.—This paper will be inserted at some length.

On the Chlorine Carbide, C_3Cl_3 .—Paul Lemoult.—A thermo-chemical paper.

Action of the Carbonic Acid of Waters upon Iron.—P. Petit.—The action of iron upon calcium bicarbonate and upon carbonic acid in solution enables us to explain the attack of iron pipes and tanks in certain waters. It gives also the mechanism of the purification of waters by iron, and the purification of syrups by iron-filings in the sugar manufacture.

Action exerted upon Solutions of Alkaline Haloid Salts by the Acids present.—A. Ditte.—On adding the acid to the solution of a neutral salt, we determine at first a decrease of the solubility, but it does not increase without limit with the quantity of acid added.

Action of Phosphorus upon Platinum.—A. Granger.—Until recently only platinum biphosphide was known. In 1884 two American chemists, Clarke and Joslin, obtained a definite compound, Pt_3P_5 , which dissolves partially in *aqua regia*, leaving an insoluble protophosphide, PtP . The dissolved matter contains the biphosphide, Pt_2P_2 . The author, on repeating this experiment, obtained a subphosphide, Pt_2P . At white redness the mass retained only 4 per cent as phosphorus.

Action on Gaseous Hydrochloric Acid upon the Alkaline Sulphites.—Albert Colson.—Experiment shows that, contrary to the opinion of some *savants* of authority, sodium sulphate, SO_4Na_2 , is attacked in the cold by dry HCl. There exist small series of tensions of hydrochloric acid gas.

The Reduction of Tungsten by Coke in the Electric Furnace.—Ed. Defacqz.—This memoir will be inserted in full.

New Instances of Normal Rotatory Dispersion.—Ph. A. Guye and P. A. Melikion.—The substances being arranged according to the increasing values of $[\alpha]_D$ we remark that the specific rotatory dispersions remain of the same order of magnitude, but they are not proportional to the specific rotatory power.

Transformation of the Sulphonated Camphophenols into Dinitro-orthocresol.—P. Cazeneuve.

On Hexa-diimediol.—R. Lespieau.

The Congelation-point of Milk.—J. Winter.—A controversial memoir in reply to MM. Bordas and Génin.

A Contribution to the Study of the Borneols and their Ethers.—J. Minguin.—These last papers are not adapted for abstraction.

Optical Analysis of Urine and Exact Determinations of the Proteids, Glucosides, and Non-fermentable Saccharoid Matters.—F. Landolph.—(See p. 25).

Revue Universelle des Mines et de la Metallurgie.
Series 3, Vol. xxxvi., No. 1.

Determination of Sulphur in the Products of Siderurgy.—The methods for the determination of sulphur in siderurgical products are classified under the following heads:—Procedures for the direct oxidation of sulphur by the moist or the dry method, followed by precipitation as barium sulphate either with or without a previous elimination of iron; procedure for direct chlorination by the dry method, and precipitation as barium sulphate; procedures by hydrogenisation of the sulphur in the dry way; procedures of evolution, H_2S being liberated by the action of dilute acids and either oxidised to form SO_3 or absorbed by saline solutions; mixed procedures. This lengthy memoir, which extends to 90 pages, requires the accompanying figures.

MISCELLANEOUS.

To Soda and Ultramarine Manufacturers.—Rud C. Gittermann writes as follows from Odessa, South Russia, under date January 8th, 1897:—"It may interest your readers that we are in great want here of manufactories of soda and ultramarine, and that competent English makers of these articles are sure to make their fortunes by establishing here. A part of the necessary capital could needs be found here. If this interests any of your readers I shall be happy to give details on enquiry."

Our Weights and Measures.—A Practical Treatise on the Standard Weights and Measures in use in the British Empire, with some account of the Metric System, by H. J. Chaney, will be published early in January. It will contain much information derived from authorised sources, and the writer's position as Superintendent of the Standards Department, Board of Trade, assures us that the information given—information now for the first time published—will be of practical use to local officers, and especially to traders generally. It is hoped also that the book will even be of use to the chemist and physicist; and that the antiquarian will find something of interest in its pages. The information given with reference to "metric" weights and measures should be of particular use to scientific authorities and to manufacturers.

MEETINGS FOR THE WEEK.

MONDAY, 18th.—Society of Arts, 8. (Cantor Lectures). "Material and Design in Pottery," by Wm. Burton, F.C.S.
Society of Chemical Industry, 8. "The Character of the London Water Supply," by W. J. Dibdin, F.I.C., F.C.S.

TUESDAY, 19th.—Royal Institution, 3. "Animal Electricity," by Prof. A. D. Waller, F.R.S.

WEDNESDAY, 20th.—Society of Arts, 8. "The Roller Boat of Mons. Bazin," by Emile Gautier.

THURSDAY, 21st.—Royal Institution, 3. "Some Secrets of Crystals," by Prof. H. A. Miers, F.R.S.

Chemical, 8. "Studies of the Properties of Highly Purified Substances—I. The Influence of Moisture on the Production of Ozone from Oxygen, and on the Stability of Ozone; II. The Behaviour of Chlorine, Bromine, and Iodine with Mercury; III. The Behaviour of Chlorine under

the Influence of the Silent Discharge of Electricity and in Sunlight," by W. A. Shenstone. "Action of Diastase on Starch," Part III., by A. R. Ling and T. L. Baker. "The Solution, Density, and Cupric Reducing Power of Dextrose, Levulose, and Invert-Sugar," by Horace J. Browne, F.R.S., G. Harris Morris, Ph.D., J. H. Millar. "Derivatives of Maclurin," Part II., by A. G. Perkin.

FRIDAY, 22nd.—Royal Institution, g. "Properties of Liquid Oxygen," by Prof. Dewar, F.R.S.

Physical, 5. "An Exhibition of some Simple Apparatus," by W. B. Croft, M.A. "On the Passage of Electricity through Gases," by E. C. Baly.

SATURDAY, 23rd.—Royal Institution, g. "Neglected Italian and French Composers," by Carl Armbruster.

ERRATA.—P. 14, col. 1, line 23 from top, for "Potass. Nitrate" read "Potass. Nitrite." P. 15, col. 2, line 33 from bottom, for "large amounts" read "larger amounts."

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1939.

TUNGSTEN HEXABROMIDE.

By HERBERT A. SCHAFFER and EDGAR F. SMITH.

THE most recent work upon tungsten bromides is that of Roscoe (*Ann. Chem.*, Liebig, clxii., 362), who endeavoured to prepare a hexabromide, but obtained instead a penta derivative from which the dibromide was subsequently made. By reference to the literature bearing upon this subject it will be noticed that bromine, diluted with carbon dioxide, was made to act upon tungsten metal exposed to a red heat. Experimental evidence is at hand that tungsten at high temperatures deoxidises carbon dioxide, thus allowing ample opportunity for the production of oxybromides, which, in spite of the greatest care, are sure to appear in larger or smaller amount. The thought also suggested itself that possibly the "red heat" at which the action was allowed to occur might have been detrimental and have indeed prevented the formation of the hexabromide.

Hence, we determined to operate in an atmosphere of nitrogen and to apply a very gentle heat to the vessel containing the tungsten. In this connection it may be mentioned that the nitrogen was conducted through a series of vessels charged with chromous acetate, sulphuric acid, caustic potash, and phosphorus pentoxide, respectively. It then entered an empty vessel, into which dry bromine was dropped from a tap-funnel, and after passing through a tall tower, filled with calcium chloride, entered a combustion tube resting in a Bunsen furnace. The anterior portion of the combustion tube was contracted at intervals, forming a series of bulbs, and at its extremity was connected with an empty Woulff bottle, followed by a calcium chloride tower, and finally a receiver filled with soda lime and broken glass. A steady current of nitrogen was conducted through this system for a period of three days. On the fourth day bromine was introduced. The tungsten contained in the combustion tube was heated very gently. In a short time brown vapours appeared. These condensed to a liquid beyond the boat and eventually passed into blue-black crystalline masses that separated from the walls of the tube, when perfectly cold, with a crackling sound. Very little heat was required to melt them, and they could with care be re-sublimed in distinct blue-black needles. The latter was collected in one of the bulbs (No. 2) previously mentioned. Other products were observed and isolated. All were analysed. Bulb No. 1—that nearest the tungsten metal—contained a black velvety compound, which upon analysis showed the presence of tungsten dibromide. Bulb No. 2 contained 0.2103 grm. of the blue-black crystals, which yielded 0.0577 grm. of tungsten, or 27.43 per cent, and 0.1543 grm. of bromine, or 73.53 per cent. The theoretical requirements of tungsten hexabromide are 27.72 per cent tungsten and 72.28 per cent bromine. The bromine percentage found is high. This may be due to traces of bromine that had not been driven out from the crystalline deposit, or to adherent silver tungstate, as some tungstic acid remained in the solution from which the silver bromide was precipitated.

A fresh portion of the blue-black crystals was prepared as before and analysed. The bromine determination was unfortunately lost. The determination of the tungsten resulted as follows:—0.4351 grm. of material gave 0.1222 grm. of tungsten, or 28.08 per cent.

A third preparation was made. On subjecting 0.1775 grm. of it to analysis these results were obtained:—

0.0496 grm. tungsten or 27.94 per cent.
0.1266 grm. bromine or 71.32 per cent.

Tabulating the series, we have:—

	Found		Mean.		Required for hexabromide.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Tungsten ..	27.43	28.08	27.94	27.81	27.72
Bromine ..	73.53	71.32	—	72.33	72.28

These figures give evidence that the body analysed is tungsten hexabromide.

In analysing the third portion of the blue-black needles the bromine was determined by placing the material in a small Erlenmeyer bulb, covering it with nitric acid and then distilling. The liberated bromine was passed into a silver nitrate solution.

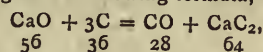
The tungsten hexabromide prepared by us consists, as already observed, of blue-black needles. Moderately elevated temperatures decompose the compound. It gives off fumes when brought in contact with the air. Water decomposes it with the formation of a royal-blue coloured oxide. Ammonia water dissolves it, the solution remaining colourless. A vapour density determination resulted negatively, as decomposition was apparent early in the experiment.—*From the Journal of the American Chemical Society*, December, 1896.

THE MANUFACTURE OF CALCIUM CARBIDE.*

By J. T. MOREHEAD and G. de CHALMOT.

(Concluded from p. 31).

WE have now still to consider a very important question, namely, how much coke and lime are necessary to produce one pound of carbide. The formation of carbide taking place according to the following formula,—



0.563 pound of carbon and 0.875 pound of calcium oxide are theoretically necessary to yield one pound of carbide. The carbide of 5 cubic feet of gas per pound contains, however, free calcium oxide. We therefore might expect that more calcium oxide and less carbon are used. In practice, however, some calcium oxide and carbon are volatilised or burned. For a succession of experiments we have weighed the mixture that was put into the furnace and that which was taken out, and analysed both. We have experienced considerable trouble in weighing the red hot material accurately, and in obtaining fair samples. We have therefore not been able to observe as to how far the consumption of calcium oxide and of carbon is influenced by the circumstances that alter the quantity and quality of the product. We found, however, that less mixture is used if properly stoked, and if the arc is kept covered. We found also that the losses of carbon are always more considerable than those of calcium oxide.

TABLE VIII.

No. of experiment.	Mixture into the furnace.		Mixture out of the furnace.	
	CaO.	C.	CaO.	C.
	Per cent.	Per cent.	Per cent.	Per cent.
1	54.70	36.32	57.01	36.89
2	54.51	34.18	57.29	33.36
3	56.23	36.46	57.00	35.62
4	55.66	36.51	55.47	35.02
5	59.70	34.43	58.16	32.94
6	56.65	36.09	55.60	32.61
7	55.44	36.32	54.93	31.09
8	50.64	34.43	51.81	34.29
9	52.08	29.39	52.09	28.90
10	49.34	33.85	41.45	28.27

* Read Sept. 3rd before the Springfield meeting of the A.A.A.S. by one of us (M.). We have made since then several additions, so as to make the article complete up to the present time. From the *Journal of the American Chemical Society*, April, 1896.

TABLE VII.

Number of experiment.	Amount of CaO put into the furnace.	Amount of C put into the furnace.	CaO obtained from the furnace.	C obtained from the furnace.	Amount of CaO used.	Amount of C used.	Production of clean carbide.	Amount of CaO per lb. of carbide.	Amount of C used per lb. of carbide.
	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lb.
1	675	448	417	270	258	178	195	1'32	0'91
2	739	463	415	242	324	221	285	1'13	0'78
3	859	557	601	375	258	182	190	1'36	0'96
4	612	402	415	262	197	140	190	1'04	0'74
5	851	491	498	282	353	209	280	1'26	0'75
6	906	577	478	280	428	297	375	1'14	0'79
7	1067	699	420	238	647	461	555	1'17	0'83
8	552	375	253	167	299	208	223	1'34	0'95
9	374	211	207	115	167	96	132	1'27	0'73
10	675	461	244	134	431	327	345	1'25	0'95
						Average		1'228	0'837

The average figures of Table VII. are rather high, for where much coke and lime have been used this is certainly partly due to losses of material by weighing into and out of the furnace, and also by insufficient stoking. In the King furnace, the under part of which shuts hermetically tight and excludes draught, and which is stoked mechanically, the amount of coke and lime necessary for making one pound of carbide will certainly be much reduced. In the figures given in Table VII. we have left the outside coating out of the calculation. In a plant where the acetylene gas is generated at once from the carbide it would pay to use this coating also for making gas. From Table VII. we see that a very large percentage of the mixture is not acted on by the arc. We have, however, reduced this amount to one-third of the mixture, and could reduce it still more without either injuring the furnace, the quantity and quality of the carbide, and without increasing the amount of carbon and lime necessary for making one pound of carbide. In the furnace used in Spray the inside is square instead of octagonal, and the dimensions are rather too large. We therefore feed more material into the furnace than is necessary.

Besides coke we have used several other carbonaceous materials for making carbide. We have used soft coal, anthracite, charcoal, pitch, tar, rosin, and asphalt, and obtained in all cases carbide. Most of these materials are not of sufficient importance to be taken into consideration, and we will only add some words about the first three.

Charcoal, owing to its small percentage of ash, yields a very pure carbide. The only drawback, besides its price, is that it is so light that the gases carry it off to a considerable amount. It is therefore necessary to add from 5 to 10 per cent more carbon to the mixture if charcoal is used than if coke is used.

We used a soft coal which contained volatile matter 19'84 per cent and ash 1'48. The mixture with soft coal gave a terrific blaze. The carbide was covered with a large amount of very porous slag, in which there was much graphite. The average of results of two runs are 6'41 pounds per horse-power in twenty-four hours and 4'33 cubic feet of gas per pound, which equals 27'75 cubic feet of gas per horse-power in twenty-four hours.

We used anthracite coal, which contained volatile matter 7'95 per cent and ash 4'02 per cent. We made two runs with slacked and two with unslacked lime. There was no appreciable difference in the use of slacked and unslacked lime. The average result of the four runs was 7'64 pounds per horse-power in twenty-four hours and 4'03 cubic feet of gas per pound, which equals 30'79 cubic feet per horse-power in twenty-four hours. These results are much lower than those obtained with coke. We cannot therefore recommend the use of either anthracite or soft coal for making carbide. The superiority of coke and charcoal over anthracite is probably due to the porosity of the former materials, which must

facilitate the volatilisation of the carbon in the electric arc, which probably must precede the formation of carbide.

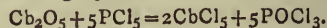
DERIVATIVES OF COLUMBIUM AND TANTALUM.*

By MARY ENGLE PENNINGTON.

(Concluded from p. 34).

Products obtained on Heating the Oxides of Tantalum and Columbium with Phosphorus Pentachloride.

ONE-HALF grm. of columbic oxide was heated with phosphorus pentachloride, the quantity being calculated from the following equation:—



The experiment was conducted in a sealed tube from which all air had been expelled, the temperature being maintained at 180—200° C., for seven hours. The resulting mass was moist, and a dirty green. The tube was opened, connected quickly with a small test-tube, and then heated in an air-bath. A small quantity of liquid distilled into the front part of the tube. This was a yellowish green, and gave with water a white precipitate, apparently a hydrated columbic oxide.

At a higher temperature, about 200° C., yellow vapours collected in the cool portion of the tube. These settled on the glass as yellow, oily drops, and on cooling solidified, long yellow needles being detected here and there. Nearly all of the substance in the tube, however, remained as the greenish mass, which had become dry. No change was observed on heating above 360° C. The tube was then wrapped in copper gauze and heated with a Bunsen lamp. The green substance swelled up, became white, iridescent, and almost filled the tube. No green colour remained. Analysis of this compound showed it to be columbium oxychloride, CbOCl_3 . The long yellow needles which had been observed in the front part of the tube changed gradually on heating, and became white and iridescent like the remainder of the substance.

This behaviour indicated the formation of a pentachloride which was then changed to oxychloride by the small quantity of air which entered the tube when it was connected with the receiver.

A second tube, heated for eight hours at 230—235° C., gave a dark yellow, semi-fluid mass. Great care was taken in this experiment to exclude all traces of moisture, and the distillation was conducted under reduced pressure. Phosphorus oxychloride in considerable quantity distilled over, leaving in the tube a yellow crystalline

* From the author's thesis presented to the University of Pennsylvania for the degree of Ph.D., 1895. From the *Journ. Amer. Chem. Soc.*, xviii., January, 1896.

substance, which, on treating with water, decomposed with hissing and an evolution of hydrochloric acid gas. This compound was analysed according to the method of Marignac (*Ann. Chim. Phys.*, viii., 5). The ignited oxide weighed 0.5642 gm. As only $\frac{1}{2}$ gm. of columbic oxide was used in the experiment, the contaminating substance was sought, and was found to be phosphorus. Two fusions with bisulphate were necessary for the extraction of this element. Phosphoric acid was also found in the filtrate from the pentoxide.

The question now arose regarding the position of this phosphorus. Is there a compound formed containing columbium, phosphorus, and chlorine, or is the phosphorus content due to an incomplete expulsion of the excess of phosphorus pentachloride?

Another experiment was therefore tried under the following conditions:—One-half gm. columbium pentoxide was heated with the calculated quantity of phosphorus pentachloride at a temperature not exceeding 210°C . for eight hours. The tube contained a yellow mass as before. It was placed in an air-bath and connected with a chlorine generator, the receiver having been previously filled with chlorine. At 190°C . a very volatile substance collected in the front part of the receiver. This was a lemon-yellow, and, when analysed, gave 15.85 per cent columbium and 6.095 per cent phosphorus.

At 190 – 200°C ., long yellow needles collected; some of these were nearly half an inch in length. Analysis gave 27.37 per cent columbium and 32.19 per cent phosphorus.

The substance, which did not volatilise at 200°C ., was brownish-yellow, and apparently crystalline. Analysis gave 28.11 per cent columbium, and 13.34 per cent phosphorus.

In none of these analyses could the chlorine content be determined, because of the violence with which water acts upon the compounds, resulting invariably in the loss of some hydrochloric acid.

It seemed probable that the brownish-yellow residue in the tube was columbium pentachloride, enclosing a small quantity of phosphorus pentachloride. To determine all three elements, the following method was used:—

The more volatile compounds having been removed by distillation in a stream of chlorine gas, the residual substance was quickly weighed and thrown into a dilute solution of silver nitrate. The precipitate of silver chloride, silver phosphate, and hydrated columbic oxide was then filtered, and washed on the filter with dilute nitric acid. The phosphoric acid obtained was determined by a magnesium mixture. Dilute ammonium hydroxide was then poured over the mixture of silver chloride and columbic oxide. It was found that all the silver salt could not be removed by this means. The mixture was therefore transferred to a porcelain crucible and reduced in a stream of hydrogen gas, the metallic silver being dissolved out with dilute nitric acid, then precipitated as chloride. The columbium remained in the form of a violet compound, which, on ignition in the air, went over to pentoxide.

A small quantity of phosphorus was obtained, which was calculated into pentachloride and deducted from the material taken.

Rose states that a columbate of silver, $\text{Cb}_2\text{O}_5\cdot\text{Ag}_2\text{O}$, is formed on the addition of silver nitrate to a solution of sodium columbate. As, upon the addition of water to columbium pentachloride, an almost perfect solution is produced for a few moments, the columbium in solution may combine with the silver. In such a case the silver chloride finally weighed would represent both the silver in combination with chlorine and that with columbium.

The analytical results are as follows:—

Substance taken = 0.8917 gm.

Phosphorus found = 0.02596 gm.

This, as phosphorus pentachloride, requires 0.14829 gm. of chlorine.

Substance taken minus $\text{PCl}_5 = 0.7175$ gm.

Columbium found = 0.2485 gm.

Columbium required = 0.2484 gm.

Chlorine found = 0.66509 gm.

Taking from this 0.14829 gm. of chlorine, which is in combination with phosphorus, we have chlorine = 0.5168 gm.; columbium pentachloride requires 0.4691 gm. Calculating the quantity of silver which, according to Rose's formula, would combine with the amount of columbium oxide found, and deducting the chlorine corresponding to it, 0.4699 gm. of chlorine is found to be in combination with the columbium.

The volatile compounds mentioned above were recalculated into phosphorus pentachloride and columbium pentachloride. It was found that, by removing the phosphorus as pentachloride, satisfactory analyses for columbium pentachloride were obtained from the residues.

Tantalalic oxide was also heated with phosphorus pentachloride, the same conditions being maintained as in the columbium experiments. A yellow mass was formed, lighter in colour than the columbium compound, and only slightly moist. The tube was placed in an air-bath, and distilled at a temperature not exceeding 245°C . This distillation was conducted under reduced pressure. A small quantity of phosphorus oxychloride distilled over, and in the front part of the tube a little phosphorus pentachloride collected. The tantalum compound remaining was light yellow, dry, and powdery—apparently amorphous. It combined with water with hissing, liberating tantalalic oxide, which contained no phosphorus. A small quantity of this element was found in the filtrate from the oxide. It was calculated into phosphorus pentachloride, and deducted from the total quantity.

Weight of substance taken = 0.6700 gm.

Weight of tantalum found = 0.3389 gm.

Required tantalum = 0.3391 gm.

Tantalum pentachloride is, therefore, formed when tantalalic oxide is heated with phosphorus as pentachloride.

Reduction of the Compounds of Columbium and Tantalum to Metal.

Two experiments aiming at the preparation of columbium and tantalum in the metallic state have been tried during this research, and I regret exceedingly that lack of time has prevented a more careful study of the reactions obtained. It is my intention to go more deeply into the subject than I have been able to do.

Experiment 1.—An iron cylinder, 3 inches in diameter, having an inch bore, was charged in the following manner:—First, a layer of dry salt, then a layer of metallic sodium, above which were placed about 7 grms. of potassium tantalum fluoride, this being followed by another layer of sodium. The cylinder was then tightly packed with dry salt, and a heavy lid screwed on. It was then placed in a wind furnace, the temperature of which was comparatively low. In less than one half-hour it was found that the cylinder had melted down, and no trace of the charge could be found.

Experiment 2.—Marignac obtained an alloy of columbium and aluminum by heating the potassium double fluoride with aluminum scales in a carbon crucible. In the experiment to be described columbic oxide was used, salt and cryolite being employed as a flux. The following layers were placed in a graphite crucible:—

1. Salt.
2. Cryolite.
3. Aluminum clippings.
4. Columbic oxide.
5. Aluminum clippings.
6. Cryolite.
7. Salt.

The proportion of these substances used were:—

2 parts C_2O_5 .
10 parts cryolite.
15 parts aluminum.
x parts sodium chloride.

The graphite lid was firmly luted on with fire-clay, the crucible was buried in a wind furnace which was kept at a white heat for eight hours. At the end of this time it was found that the graphite crucible had been severely attacked. It was reduced to a shapeless mass, but on breaking a powdery substance was found, in which were contained many little metallic buttons varying in size from a large pea to those of microscopic proportions. These were carefully picked out, and various reagents tried upon them.

Single acids do not attack them. Aqua regia makes a slight impression on long heating. Fusion with bisulphate affords only a partial decomposition. The substance is exceedingly light; it is dark grey, and does not alter in the air. A partial oxidation occurs after prolonged heating in the air. The substance is not brittle.

Summary.

1. The decomposition of columbite is more readily and satisfactorily accomplished by the Gibbs than by the bisulphate method. This method is also more valuable for the preparation of large quantities of pure oxides.

2. The qualitative reactions of columbium, tantalum, and titanium, when existing as double fluorides, are not the same as when the metals exist as tantalates, columbates, and titanates.

3. The action of the electric current upon tantalum and columbium double fluorides gives a lower hydrated oxide. The precipitation is not complete.

4. It was hoped that in preparing the double fluorides of columbium, tantalum, and titanium with rubidium and caesium, a difference in solubility of the salts would be found which would afford a better separation of these metallic oxides under discussion. This hope has not been realised.

5. Heating the oxides of columbium and tantalum in sealed and vacuous tubes with phosphorus pentachloride yields the pentachlorides of these metals and phosphorus oxychloride.

I take pleasure in acknowledging the kindness shown, and the interest taken in the preceding work, by Dr. Edgar F. Smith, of the University of Pennsylvania, in whose laboratory it was carried out.

DETERMINATION OF THE ATOMIC MASSES OF SILVER, MERCURY, AND CADMIUM, BY THE ELECTROLYTIC METHOD.*

By WILLETT LEPLEY HARDIN.

(Continued from p. 29).

FIRST SERIES.

Experiments on Silver Nitrate.

THE nitrate of silver seems to fulfil the conditions necessary for accurate analyses, inasmuch as it is stable and crystallises in well-defined crystals, which can be fused without decomposition.

Preparation of Silver Nitrate.

The material used in these experiments was prepared by dissolving pure silver in pure aqueous nitric acid in a porcelain dish. An excess of silver was used, and after complete saturation the solution was poured off from the metal into a second dish; and evaporated to crystallisation. The perfectly transparent rhombic plates of silver

nitrate which separated were dissolved in pure water and re-crystallised. The crystals were then carefully dried, placed in a platinum crucible which rested in a larger platinum dish, and gradually heated to fusion. After cooling, the perfectly white opaque mass was broken up and placed in a ground-glass stoppered weighing-tube, and kept in a desiccator in a dark place.

Mode of Procedure.

The platinum dish in which the deposit was made was carefully cleaned with nitric acid and dried to constant weight. It was then placed in a desiccator over anhydrous calcium chloride, and this, together with the desiccator containing the tube of silver nitrate, was placed in the balance room, where they were allowed to remain until their temperatures were the same as that of the room. After weighing the platinum dish, the tube of silver nitrate was weighed and part of the salt removed to the dish, after which the tube was re-weighed. The difference in the two weighings, of course, represented the weight of silver nitrate used in the experiment. Enough water to dissolve the nitrate was added to the dish, and then a solution of potassium cyanide, made by dissolving 75 grms. of pure potassium cyanide in 1 litre of water, was added until the silver cyanide first formed was completely dissolved. The dish was then filled to within a quarter of an inch of the top with pure water, and the solution electrolysed with a gradually increasing strength of current. The following Table will show the strength of current and the time through which it acted:—

Time of action.	Strength of current.
2 hours.. ..	N.D ₁₀₀ =0.015 ampères.
4 "	N.D ₁₀₀ =0.030 "
6 "	N.D ₁₀₀ =0.075 "
4 "	N.D ₁₀₀ =0.150 "
4 "	N.D ₁₀₀ =0.400 "

By gradually increasing the strength of current in this way the silver came down in a dense white deposit. When the deposition was complete, before interrupting the current, the liquid was syphoned from the dish, pure water being added at the same time. This was continued until the cyanide was completely removed. The dish with the deposit was washed several times with boiling water, and carefully dried. It was then placed in a desiccator, and allowed to remain in the balance room until its temperature was the same as that of the room, when it was re-weighed.

Weight of platinum dish = 71.27302 grms.

Weight of silver nitrate = 0.31198 gm.

Temperature, 22°.

Barometric pressure, 770 m.m.

Weight of platinum dish + silver deposit = 71.47104 grms.

Temperature, 22°.

Barometric pressure, 760 m.m.

Density of silver nitrate = 4.328.

" brass weights = 8.5.

" platinum dish = 21.4.

" metallic silver = 10.5.

" atmosphere at the time of weighing the empty dish and silver nitrate = 0.001212.

" atmosphere at the time of weighing the platinum dish + silver deposit = 0.001196.

Computing on this basis we have the following:—

$$0.31198 \left(1 + \frac{0.001212}{4.328} - \frac{0.001212}{8.5} \right) = 0.31202 = \text{weight of } \text{AgNO}_3 \text{ in vacuo.}$$

$$\frac{71.27302 - \left(1 + \frac{0.001212}{21.4} - \frac{0.001212}{8.5} \right)}{1 + \frac{0.001196}{21.4} - \frac{0.001196}{8.5}} = 71.27291 = \text{wt. of platinum dish at } 22^\circ \text{ and } 760 \text{ m.m.}$$

* Contribution from the John Harrison Laboratory of Chemistry No. 13. From the author's thesis presented to the Faculty of the University of Pennsylvania for the degree of Ph.D.—From the *Journal of the American Chemical Society*, xviii., p. 990.

$71'47104 - 71'27291 = 0'19813$ = weight of deposit at 22°
and 760 m.m.

$$0'19813 \left(1 + \frac{0'001196}{10'5} - \frac{0'001196}{8'5} \right) = 0'19812 = \text{weight of deposit in vacuo.}$$

Taking O = 16 and N = 14'04, the atomic mass of silver

$$= \frac{0'19812 \times 62'04}{(31202 - 19812)} = 107'914.$$

Ten observations on silver nitrate computed in the foregoing manner are as follows:—

	Weight of AgNO ₃ . Grms.	Weight of Ag. Grm.	Atomic mass of silver.
1	0'31202	0'19812	107'914
2	0'47832	0'30370	107'900
3	0'56742	0'36030	107'923
4	0'57728	0'36655	107'914
5	0'69409	0'44075	107'935
6	0'86367	0'54843	107'932
7	0'86811	0'55130	107'960
8	0'93716	0'59508	107'924
9	1'06179	0'67412	107'907
10	1'19849	0'76104	107'932
Mean	= 107'924		
Maximum ..	= 107'960		
Minimum ..	= 107'900		
Difference ..	= 0'060		
Probable error	= $\pm 0'005$		

Computing the atomic mass of silver from the total quantity of material used and metal obtained, we have 107'926.

(To be continued).

INTERNATIONAL EXHIBITION AT BRUSSELS, 1897.

THE rage for those displays known as "international expositions" still continues. Regardless of the fact that few persons are benefitted by such gatherings, except railways, hotel proprietors, and the small but noisy class who might be called professional exhibitionists, most States still think it their duty or their policy to challenge the world to an industrial competition. This season Belgium holds the arena. Its Exhibition will include a section of the Sciences divided into the following seven classes: Mathematics and astronomy, physics, chemistry, geology and geography, biology, anthropology, and bibliography. Participators will have nothing to pay for space, and will have to pay reduced freights for the conveyance of their exhibits.

We cannot, however, here refrain from reminding ourselves that any and every advance or improvement in any science will not fail to have become known to the world through the scientific and technical press, so that the communications and exhibits at such gatherings will have lost much of their novelty. We have much pleasure in admitting that there is here no class for political economy, which, whenever tolerated, slides inevitably into party politics with its usual amenities. The Belgian Government proposes a series of questions, and offers for the best solutions prizes amounting to the modest aggregate of 20,000 francs. Pamphlets giving full particulars may be obtained on application to the General Commissariat of the Government, at 17, Rue de la Presse, Brussels.

Among the problems to be solved in the chemical class must be mentioned—

To establish the constitution of camphor by means of reactions, both analytical and synthetic, to differentiate the optical isomers by means of new chemical reactions.

A practical method for transforming without great expense atmospheric nitrogen into ammonia.

A practical method for the preparation of chlorine from CaCl₂, more economical than those already in use.

A new process for fixing the azo-colour upon the various textile fibres preferable to those hitherto known.

The acid HI being not easy to 'prepare,' to find an essentially practical method for its production.

An improvement in the procedures of fractionated distillation.

Under biology there is the demand for new researches of living beings by means of the X rays.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY
SAMPLES OF THE WATER SUPPLIED TO LONDON
FOR THE MONTH ENDING DECEMBER 31ST, 1896.

By WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolis Water Act, 1871.

London, January 11th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 175 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Dec. 1st to Dec. 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in a previous report.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 175 samples examined all were found to be clear, bright, and well filtered.

Rain has fallen at Oxford on almost every day during the month, there being but nine days when none was recorded; the average fall for the month of December is 2'10 inches, the actual fall this month has been 3'13 inches, giving an excess of 1'03 inches. The deficiency for the whole year amounts to 3'53 inches, or 13'3 per cent, on a thirty years' average of 25'72 inches.

The details of the rainfall are shown in the following table:—

Rainfall in Inches at Oxford, Month by Month, during
the Year 1896.

	Actual fall.	Mean of 30 years.	Difference from the mean.	
January ..	0'63	2'16	- 1'53	—
February ..	0'36	1'76	- 1'40	—
March ..	2'45	1'50	—	+0'95
April ..	0'58	1'66	- 1'08	—
May ..	0'20	1'83	- 1'63	—
June ..	2'42	2'11	—	+0'031
July ..	1'40	2'68	- 1'28	—
August ..	2'01	2'32	- 0'31	—
September..	5'47	2'43	—	+3'04
October ..	2'85	2'75	—	+0'10
November..	0'79	2'42	- 1'63	—
December..	3'13	2'10	—	+1'03
	22'29	25'72	- 8'86	+5'43

Month.	Thames, unfiltered.	Five Thames- derived Companies filtered.	New River unfiltered.	New River filtered.	River Lea, unfiltered.	River Lea (East London), filtered.
January	1824	36	1525	31	2005	24
February	1453	26	1368	29	1109	28
March	2160	41	1938	27	1900	29
April	1833	33	1501	17	1643	34
May	1301	21	950	25	1216	73
June	2081	32	836	35	1729	28
	1775	31	1353	27	1600	36 Mean of 1st 6 months.
July	2731	21	1886	22	2859	33
August	1133	17	361	3	537	5
September	1331	11	709	4	1642	14
October	2691	19	671	4	742	14
November	2223	21	1054	20	1178	6
December	4613	60	2729	57	1825	15
	2453	24	1235	18	1464	14 Mean of 2nd 6 months.
	2114	27	1294	22	1532	25 Mean of 12 months.

It will be seen that the large excess of 3'04 inches in September has been mainly instrumental in reducing a deficiency which was becoming very serious.

Our bacteriological examinations of the waters during the month of December give the following results:—

	Colonies per c.c.
Thames water, unfiltered	4613
Thames water, from the clear water wells of the five Thames-derived supplies.. highest	248
Ditto ditto lowest	6
Ditto ditto .. (13 samples) mean	60
New River water, unfiltered	2729
New River water, from the Company's clear water well	57
River Lea water, unfiltered	1825
River Lea water from the East London Com- pany's clear water well	15

The accompanying table shows the bacterial variations during the year.

These results show that, taking the whole year's bacteriological examinations, the high efficiency of the filtration and the excellent character of the London waters have been uniformly maintained.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.
JAMES DEWAR.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, December 17th, 1896.

Mr. A. G. VERNON HARCOURT, President, in the Chair.

MESSRS. Alexander Scott, Frederick B. Power, W. W. Cobb, and Claude M. Thompson were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Alfred Cartmell, Alexandra Road, Burton-on-Trent; William Diamond, Pye Bridge, Alfreton; William Buckland Edwards, 5, Garlinge Road, Brondesbury, N.W.; Vaughan Harley, M.D., 25, Harley Street, W.; Fred Ibbotson, B.Sc., 9, Melbourne Road, Spring Vale, Sheffield; David Smiles Jerdan, M.A., B.Sc., 68, Union Street, Greenock; Edward Rosling, Melbourne, Chelmsford; Henry Potter Stevens, B.A., 14, Lower Sloane Street,

Chelsea, S.W., Harry Thompson, Walton House, West Parade, Anlaby Road, Hull.

The certificate of the following candidate, recommended by the Council, under Bye-law I., par. 3, was also read:—Jyoti Bhushan Bhaduri, Presidency College, Calcutta.

Of the following papers those marked * were read:—

*165. "On the Experimental Methods employed in the Examination of the Products of Starch-hydrolysis by Diastase." By HORACE T. BROWNE, F.R.S., G. HARRIS MORRIS, Ph.D., and J. H. MILLAR.

The paper is divided into the following sections: (1) the determination of solids from solution-density; (2) determination of specific rotary power; (3) the relation of $[\alpha]_D$ to $[\alpha]_D$; (4) determination of cupric reducing power; (5) limits of accuracy of the methods.

The authors state that this account is a preface to a series of papers dealing with the question of starch-hydrolysis, and is a critical review of the experimental methods which have been employed by different observers who have approached this subject. An attempt has also been made to remove the misunderstanding which still exists as to the relations of the different systems of notation.

The determination of the total solids from the density of the solution by the employment of the "divisor" method admits of great accuracy if the solution-densities of the pure substance have been previously determined.

The "divisors" at varying concentration have been determined for cane sugar, maltose, dextrose, levulose, soluble starch, and the mixed products of starch-hydrolysis of various grades, and the results have been plotted out in the form of curves whose equation is given in each case. The pure substances used in constructing these curves were dried in a vacuum over phosphoric pentoxide at temperatures from 100° to 130°.

For mixed starch hydrolytic products the divisor for equal concentrations increases with the specific rotary power, and in such a regular manner that when the value of R is known, the divisor at any given concentration can be calculated. From the relation which this divisor bears to the divisor of the apparent maltose present in the mixed hydrolytic products, it is deducible that the divisor for the amylin constituent is constant for equal concentrations, even in starch products of very different grades of hydrolysis.

In the section on specific rotary power, the methods of exact determination are discussed, and the relations of $[\alpha]_D$, $[\alpha]_{13.86}$, and $[\alpha]_D$ are defined for substances of equal dispersive power. As the dispersive power of cane sugar is sensibly different from that of dextrose and starch hydrolytic products obtained by diastase, the factors for the

conversion of $[\alpha]_D$ into $[\alpha]_D$ are not identical in these cases. Much confusion of these relations has also been introduced by the unrecognised fact that $[\alpha]_D$ has been referred to two distinct rays in the yellow of different refrangibility.

The cupric-reduction of maltose and of the products of starch transformation is constant only when the conditions of experiment are identical. These are exactly defined for the authors' method of procedure, and the reducing values are given in tabular form, and are compared with those of other observers.

*166. "On the Specific Rotation of Maltose and of Soluble Starch." By HORACE T. BROWN, F.R.S., G. HARRIS MORRIS, Ph.D., and J. H. MILLAR.

The authors' determinations of the specific rotatory power of maltose at a temperature of 15.5° do not confirm the statement of Meissl that the values of $[\alpha]_D$ vary with the concentrations between 2 and 20 per cent, but confirm the general statement of Ost that between these limits the specific rotatory power is constant. At higher concentrations than 20 per cent the specific rotatory power diminishes slightly.

The actual results point to a value of $[\alpha]_D = 137.93^\circ$, which is sensibly greater than Ost's value of 137.46° at 15.5° .

This discrepancy is due to the fact that Ost employed weighed quantities of hydrated maltose which had been dried in a desiccator over sulphuric acid. The authors find that even after six weeks' drying in this manner, hydrated maltose contains 0.46 per cent more water than corresponds to $C_{12}H_{22}O_{11} \cdot H_2O$. If Ost's numbers are corrected for this they give values, up to 20 per cent concentrations, of $[\alpha]_D = 138.12^\circ$ at 15.5° , a result almost exactly identical with that of the authors.

The specific rotatory power of soluble starch for concentrations of 2.5 to 4.5 per cent is at 15.5° , $[\alpha]_D = 202.0^\circ$.

*167. "On the Relation of the Specific Rotatory and Cupric-reducing Powers of the Products of Starch-hydrolysis by Diastase." By HORACE T. BROWN, F.R.S., G. HARRIS MORRIS, Ph.D., and J. H. MILLAR.

When starch is transformed by diastase, a certain relation is always found to subsist between the cupric reduction and specific rotatory power of the hydrolytic products. This relation can be expressed in such a manner as to be entirely independent of any view we may hold as to the true nature of the transformation products, and it is of so exact a nature that if one property is known the other can be predicted with certainty. This is true not only for the mixed hydrolytic products, but for any fractionated portion of them.

The authors regard this fact as lying at the root of the whole question of starch hydrolysis, and, as it is still not admitted by most continental workers, they bring forward a large amount of fresh evidence which they regard as absolutely conclusive.

The results of the examination of 70 different starch transformations are given, some of them mixed products, others fractionated products, the specific rotatory and cupric-reducing powers being given in the various notations in use. When the experimental results are plotted on a system of rectangular co-ordinates, the degrees of specific rotation between soluble starch and maltose being represented on the line of ordinates, and the cupric reducing powers from soluble starch to maltose on the line of abscissæ, the values all fall practically on a straight line joining the points of intersection of the co-ordinates corresponding to the optical and reducing properties of soluble starch and of maltose respectively.

The properties of soluble starch being $R=0$, $[\alpha]_D = 202^\circ$, and of maltose, $R=100$ and $[\alpha]_D = 138.0^\circ$, then the relation of specific rotation and cupric reduction for any mixture or fractionation of the starch-hydrolytic products will be expressed by $[\alpha]_D = 202 - 0.64 R$.

The differences in the calculated and observed values

for the 70 cases of hydrolysis examined are given, and are shown to be very small indeed.

The authors have examined the published results of C. J. Lintner and of Ost, both of whom have denied the existence of any relation between $[\alpha]_D$ and R , and find that, when rightly interpreted, they, for the most part, strictly conform to the law of relation expressed above.

DISCUSSION.

Dr. ARMSTRONG, after commenting on the value of the information brought under the notice of the Society by Mr. Horace Brown and his co-workers, and on the remarkable accuracy with which starch could now be estimated, expressed the hope that it would be possible ere long to determine what really took place when starch was hydrolysed; he thought it was time that we should no longer be content merely to determine certain analytical factors; we ought rather to seek for chemical methods which would render it possible to separate and isolate the products.

Mr. A. R. LING asked what value the authors found for the cupric reducing power of maltose when Wein's method was used.

Dr. G. H. MORRIS, in reply, said that they found that Wein's tables give results about 5 per cent too low when the cupric reduction of maltose is estimated by Wein's method, and the copper obtained calculated into maltose by the table; in other words, perfectly pure maltose gives $R=95-96$ instead of 100.

*168. "The Action of Hydrogen Peroxide and other Oxidising Agents on Cobaltous Salts in presence of Alkali Bicarbonate." By R. G. DURRANT, M.A.

Similar green solutions may be obtained by adding hydrogen peroxide, sodium hypochlorite, chlorine, bromine, or ozone to cobaltous salts in presence of alkali bicarbonates—or by adding a cobaltous salt to the anode of previously electrolysed potassium carbonate.

The green colour is not destroyed by excess of cold acetic acid, but is rendered rather bluer in tint. This acetic solution is reduced by hydrogen peroxide.

The evidence so far obtained shows (1) that the cobalt is in the "cobaltic state." This is proved by the results of three volumetric methods—in which standard sodium hypochlorite, hydrogen peroxide, and sodium sulphite are respectively employed—green precipitates, produced from the green solutions, gave results showing that the available oxygen closely approximates to that to be expected from cobaltic hydrate.

(2) That the green colour of the solutions and of the precipitates appears not to be due to a particular alkali, since (i.) identical tints were obtained with the five different alkali bicarbonates, (ii.) potassium cobaltic nitrite gives no green colour with bicarbonates, (iii.) green precipitates washed free from all alkali, and digested with cold weak acetic acid, give green filtrates.

(3) That carbon dioxide is necessary both for the formation and preservation of the green colour. The green colour of the acetic solution remains only so long as carbon dioxide is present. The green precipitates (free from alkali) retain carbon dioxide so long as they remain green, and lose it when they become brown. It is, therefore, possible that the green cobaltic compound is of the nature of a carbonate.

DISCUSSION.

Several speakers, including the PRESIDENT, expressed the view that whilst the author had made it clear that the green substance was a cobaltic compound, further proof was needed of the suggestion that the salt formed was a cobaltic carbonate.

Dr. RIDEAL mentioned that sodium peroxide, as well as hydrogen peroxide, gave rise to the green colour, provided that an alkali bicarbonate was also present.

Dr. ARMSTRONG said that he would like to give expression to the opinion that the time was come to determine what should be their course of action with regard to the publication of the discussions that took place at the meetings; of late there had been an almost entire absence from the Proceedings of reports of the remarks made in the room, although these had often been of a nature which made it desirable that they should be brought under the notice of the Fellows generally. If the secretaries could not undertake the work, steps should be taken to procure a proper report. Personally he had had no difficulty in obtaining reports during the nine years in which he had charge of the Proceedings, and he did not believe that there would be any difficulty. Without such reports the Proceedings were of little value.

Professor DUNSTAN said that Fellows attending the meetings were aware that it was not often that a comprehensive discussion followed the reading of an ordinary paper. All important remarks and suggestions made at the meetings had been recorded in the Proceedings, and although no attempt had been made to record everything, and there might occasionally be room for difference of opinion as to what was important, he was always glad to receive from speakers, after the meeting, reports of their remarks, which he believed had been in nearly every case inserted in the Proceedings. He had, however, not thought it desirable to print Dr. Armstrong's remarks, of the omission of which Dr. Armstrong now complained, made on two recent occasions, proposing to record the time occupied by readers of papers. The method adopted by the speaker's predecessor in office in reporting discussions had given rise to much dissatisfaction. If the present plan was not thought sufficient, then a shorthand report of the discussions could be taken. As a matter of fact, however, the main value of the Proceedings lies in its being the means of bringing at an early date under the notice of the Fellows, not merely remarks and suggestions made at the meetings, but concise abstracts of the papers read, the full publication of which could not take place in the Journal until much later.

The PRESIDENT remarked that if a full report of the proceedings were considered desirable, its preparation could not be included in the duties of the Honorary Secretaries. He was disposed to think, however, that if it were generally known that the Secretaries were ready to receive from speakers after the meeting a few sentences giving the substance of their remarks, that this would meet the case in nearly every instance.

169. "Electrical Conductivity of Diethylammonium Chloride in Aqueous Alcohol." By JAMES WALKER, Ph.D., D.Sc., and F. J. HAMBLY, F.I.C.

The authors have determined the conductivity of diethylammonium chloride dissolved in pure water, and in 10·1, 30·7, 49·2, 72·0, 90·3, and 99·0 per cent alcohol, by volume, at dilutions ranging from 10 litres to 8000 litres. Tables and curves have been constructed, showing the variation of the molecular conductivity and the degree of dissociation with varying dilution and varying proportions of alcohol.

170. "Formation of Substituted Oxytriazoles from Phenylsemicarbazide." By GEORGE YOUNG, Ph.D., and HENRY ANNABLE.

The action which takes place when a mixture of phenylsemicarbazide and benzaldehyde is oxidised, has been re-investigated, and the views expressed by one of the authors in a previous paper (*Trans.*, 1895, lxvii., 1063) have been confirmed. The following aldehydes yield oxytriazoles by this action: metanitrobenzaldehyde, paranitrobenzaldehyde, metatoluic aldehyde, terephthalic aldehyde, cinnamic aldehyde.

The authors have failed to obtain oxytriazoles from formaldehyde, acetaldehyde, paraldehyde, isobutyric aldehyde.

171. "α-Bromocamphorsulpholactone." By C. REVIS and F. STANLEY KIPPING, Ph.D., D.Sc.

When α-bromocamphor is treated with anhydrosulphuric acid, or with chlorosulphonic acid, it is converted into α-bromocamphorsulphonic acid (*Trans.*, 1893, lxiii., 548). In the course of some experiments on the preparation of this sulphonic acid, it was found that when 70 per cent anhydrosulphuric acid is added to a solution of α-bromocamphor in chloroform, the product consists, to some extent, of a crystalline compound which is insoluble in water.

This substance has the composition $C_{10}H_{13}BrSO_4$ (found C=38·8, H=4·3, Br=25·1, S=9·7 per cent.; calculated C=39·0, H=4·2, Br=25·8, S=10·3 per cent). It appears to be a bromocamphorsulpholactone, and its formation is doubtless due to the oxidation of hydrogen to hydroxyl accompanying sulphonation, water being then eliminated from the hydroxysulphonic acid; it is, probably, closely related to the dibromocamphorsulpholactone, $C_{10}H_{12}Br_2SO_4$, recently described (Lapworth and Kipping, *Proc.*, 1896, xii., 77), and it resembles the latter in ordinary properties. It crystallises from chloroform and ethylic acetate in lustrous transparent plates or prisms, melts at about 290°, and is moderately easily soluble in boiling acetic acid, chloroform, and ethylic acetate. It is very stable, and separates, unchanged, from a solution in nitric acid (sp. gr. 1·4), even after heating for some time; it seems not to be attacked by cold potash (sp. gr. 1·3), and, even on boiling, it is only slowly dissolved.

Dr. Lapworth has, independently, observed the formation of this lactone from α-bromocamphor and anhydrosulphuric acid.

172. "Dimethylketo-hexamethylene." By F. STANLEY KIPPING, Ph.D., D.Sc.

In a recent paper on camphoric acid (*Amer. Chem. Journ.*, 1896, xviii., 685), Noyes describes the preparation, from dihydrocampholytic acid, of a ketone which forms an oxime melting at 112–113°, and possesses an odour similar to that of camphoroxime. On comparing the melting point of this oxime with that of the isomeric oxime of dimethylketo-hexamethylene, he found that, for the latter, the author had given the melting point 114–115° (*Trans.*, 1895, lxvii., 349), whereas Zelinsky had given it as 104–105° (*Ber.*, 1895, 28, 781). Noyes himself then prepared dimethylketo-hexamethylene oxime, and found the melting point to be 120–122°.

The possible identity of the two oximes in question being a matter of great importance—for, if their identity were established, much light would be thrown on the constitution of camphor—the author has prepared dimethylketo-hexamethylene by the improved method recently described (Kipping and Edwards, *Proc.*, 1896, xii., 188), and has made further experiments with this substance.

The oxime, prepared in the usual manner, is at first very oily, apparently from the presence of unchanged ketone, but it soon becomes a semi-solid crystalline mass; when freed from oil and re-crystallised once or twice, it melts quite sharply at about 114°, but further purification raises the melting point to 117·5° (uncorr.), at which point it remains, even after six successive crystallisations from different solvents. This melting point and that previously recorded were taken with an ordinary standard thermometer; observations made with a short thermometer, the thread of which was entirely immersed, gave a m. p. of 118·5–119°. Noyes does not state whether the m. p. 120–122° is corrected, nor how the observation was made, and the range of 2° would seem to indicate that the substance did not melt sharply; he also leaves the identity of his dimethylketo-hexamethylene oxime with the oxime of the ketone which he obtained from camphor an open question.

Noyes suggests that the several preparations of the oxime obtained respectively by Zelinsky, by himself, and by the author, may be mixtures of stereoisomerides, and the latter has therefore directed attention to this possi-

bility; there are certainly indications of the presence of more than one substance in the crude oxime, as a few crystals, melting not sharply at about 75° , have been separated; nevertheless, the only crystalline product which has yet been isolated in any quantity is that which melts sharply and constantly at $118.5-119^{\circ}$ (corr).

This oxime crystallises from a mixture of chloroform and light petroleum in lustrous transparent prisms, which have been examined by Mr. Pope. "The crystals consist of monosymmetric prisms, which show the forms {100}, {001}, {110}, and {111}; the plane of symmetry is the optic axial plane, and an optic axis emerges normally to the face (100). Some faces give good reflections, but parallel faces do not give images at 180° to one another, a behaviour which is frequently observed in the case of mixtures." This indication that the oxime may be a mixture, in spite of its constant melting point, must be borne in mind, and if confirmed, the different melting points of the various preparations would be accounted for.

In order to facilitate the identification of dimethylketohexamethylene, the author has prepared the semicarbazone; this compound slowly separates in crystals on warming the ketone with a solution of semicarbazone hydrochloride and sodium acetate in dilute alcohol. After re-crystallisation it melts at about 196° , and further treatment does not seem to change its melting point. A sample dried at 100° gave C=59.26, H=9.36 per cent; calculated for $C_6H_{17}N_3O$, C=59.02, H=9.29 per cent.

Dimethylketohexamethylene semicarbazone is fairly soluble in cold chloroform, but less so in cold benzene and ethylic acetate, and crystallises best from methyl alcohol in the form of small translucent well-defined prisms. Heated slowly from about 175° , and using a short thermometer, it begins to sinter at about 190° , and melts completely at about $200-201^{\circ}$, effervescing, but not darkening; the m. p. depends on the size of the crystals and on the rate of heating. The crude semicarbazone seemed to be homogeneous, and the yield appeared to be good, but as, on re-crystallising the preparation from boiling acetic acid, most of it suffered decomposition, further experiments are necessary to prove that only one semicarbazone exists.

173. "The Localisation of Deliquescence in Chloral Hydrate Crystals." By WILLIAM JACKSON POPE.

Chloral hydrate crystallises from solution in large monosymmetric plates, showing the forms {100}, {011}, and {111}, and having the axial ratios—

$$a : b : c = 1.6369 : 1 : 1.3951, \beta = 59^{\circ} 5';$$

these crystals consist of the same modification of chloral hydrate as was obtained in previous experiments (Pope, *Proc.*, 1896, xii., 142), and described as the biaxial modification, stable at ordinary temperatures. The crystals deliquesce in the air, but in a peculiar manner; the forms {011} and {111} rapidly absorb water vapour, and after a few minutes exposure become covered with a layer of solution, whilst the faces of the form {100} remain perfectly bright during a considerable time. The attraction for moisture exercised by the pinacoid {100} is thus much less than that exhibited by the other two forms.

It is consequently concluded that crystal deliquescence, like crystal solubility and other properties, varies with the direction in the crystal perpendicular to which its intensity is measured.

174. "Enantiomorphism." By WILLIAM JACKSON POPE and FREDERIC STANLEY KIPPING.

Crystals of the two enantiomorphous forms of a substance which exhibits circular polarisation only in the crystalline state, and in which the circular polarisation is an inherent property of the crystal structure, i.e., of a substance belonging to Class 2b (Pope, *Trans.*, 1896,

lxix., 971), should be deposited from the optically inactive solution in equal numbers, unless any disturbing factor is operative favouring the deposition of crystals of one particular enantiomorphous form, as, for example, contact of the slightly supersaturated solution with a crystal of that form. The truth of this statement can be demonstrated from our present knowledge of crystal structure, and is also evident from a consideration of the recent work of Landolt (*Ber.*, 1896, xxix., 2404), who showed that the crystalline powder of sodium chlorate, which rapidly separates from aqueous solution, consists of almost equal quantities of dextro- and lævo-rotatory crystals. The authors have extended these observations, and by taking a number of different crops of the large crystals deposited by spontaneous evaporation of sodium chlorate solution, have ascertained that the average numbers of dextro- and lævo-crystals deposited are the same, in absence of any disturbing factor.

It seemed probable that if a substance which is optically active in solution is introduced into an aqueous solution of sodium chlorate, the presence of the former would favour the deposition of chlorate crystals of one particular enantiomorph, and experiments were consequently made to test this view. About 5 per cent of some substance, such as dextrose, mannitol, and isodulcitol, was dissolved in a saturated sodium chlorate solution, and the crystals of the salt deposited on spontaneous evaporation examined; a great preponderance of lævo-crystals separated from the dextrose solutions whilst in the separation from the isodulcitol solutions the dextro-crystals were in excess. The mannitol solutions deposited rather more lævo than dextro-crystals; a number of crops from each solution were collected, and similar behaviour was noticed with each crop.

This selective deposition would seem to indicate, as would, indeed, be expected, from a consideration of the equilibria possible in such systems, that the solubility of a dextro-enantiomorph of Class 2b (see above) in a liquid containing an optically active substance, differs from the solubility of the lævo-enantiomorph in the same solvent. Solubility determinations, and also determinations of the rates of growth of dextro- and lævo-crystals of sodium chlorate in optically active solutions are in progress.

There would seem to be no *a priori* reason why a substance optically active in solution only and possessing a high specific rotation, should exert more directive influence on the deposition of crystals of Class 2b than an optically active substance of very low specific rotation, the only condition necessarily favouring the deposition of crystals of a particular enantiomorph being that there should be an asymmetric compound in solution. Using methods such as those indicated above, it might, therefore, be possible to determine with ease and rapidity whether certain substances which, although containing asymmetric carbon atoms, are optically inactive in solution, are really asymmetric compounds, the inactivity in solution being due to a compensation brought about amongst the four different groups attached to one asymmetric atom. Experiments respecting this point are in progress.

Several cases, such as that of camphorsulphonic chloride (Kipping and Pope, *Trans.*, 1893, lxiii., 560), are known in which equal quantities of optical antipodes, when crystallised together, apparently do not form a racemic compound. In the light of the foregoing results it should be possible to effect a partial separation of such mixtures, and even of racemic compounds, by crystallising them from a solution containing an optically active substance. Experiments on the separation of a number of racemic compounds, and of inactive mixtures of optical antipodes by methods based on the above considerations, have been commenced, but the results are not yet sufficiently conclusive to warrant any definite statements respecting them.

Premising the truth of the considerations stated above, Eakle's observation (*Zeit. f. Kryst.*, 1896, xxvi., 562) that

a sodium periodate solution containing sodium nitrate deposits more lævo- than dextro-crystals of the periodate, is quite incomprehensible.

Important Notice to Authors of Papers.

The attention of authors is directed to the following resolution of the Council.

"No title shall be included in the list of titles of papers to be brought before a Meeting of the Society, unless the paper and an abstract of it are in the hands of the Secretaries at least three days before the date of the Meeting; and no announcement of titles can be made in the Proceedings until the papers have been received by the Secretaries."

NOTICES OF BOOKS.

Register of the Associates and Old Students of the Royal College of Chemistry, the Royal School of Mines, and the Royal College of Science, with Historical Introduction and Biographical Notices and Portraits of Past and Present Professors. By THEODORE G. CHAMBERS, Assoc. R.S.M. London: Hazell, Watson, and Viney, Limited, 1896. 8vo., pp. 230.

THE "Department," as it is often styled in brief, is not by any means sufficiently known either to its admirers or to its critics. Its origin, its development, its past history, and its future prospects are all debatable subjects.

The first step towards the formation of the establishment in question was taken in 1832 by Sir Henry de la Beche. He suggested that a collection should be formed and placed under the charge of the Office of Works. The institution thus founded was named the Museum of Economic Geology, and was to contain specimens of various mineral substances used for roads or constructing public works or buildings, employed for useful purposes or from which useful metals are extracted.

The building first occupied was No. 6, Craig's Court, and Richard Phillips, F.R.S., an eminent analyst, was appointed curator. In the laboratory of the Institution samples of ores, soils, and general minerals were analysed at a fixed moderate charge. The establishment was transferred from Craig's Court to Jermyn Street in 1849, and during the following years boxes and hampers which had been lying for years at Craig's Court were opened and their contents classified and arranged. In 1851 the formal opening of the museum took place under the chairmanship of Prince Albert. In 1854 Huxley was appointed Professor of Natural History, *vice* E. Forbes, who had accepted the Chair of Natural History at the University of Edinburgh. Robert Hunt resigned the lectureship on physical science, and Professor Sir G. G. Stokes was appointed in his stead. It must be mentioned that Prof. Forbes, F.R.S., in an introductory lecture on the "Educational Uses of Museums," commented rather sarcastically on the apathy evinced by the public.

With the inauguration of the "Department" a change ensued. Whereas the Director-General of the Geological Survey and Director of the School of Mines had hitherto reported direct to a Minister of State, he had now to conduct his communications through Mr. Henry Cole, who, in 1854, was constituted Inspector-General of Schools and Museums, his title being afterwards changed to Secretary of the Department of Science and Art and Director of the South Kensington Museum.

Lyon Playfair resigned his appointment in 1858, and Captain F. Donnelly virtually obtained the executive control of the Department of Science and Art, a striking feature being the preponderating military character of the "Department." It is sarcastically said abroad that Britain puts her army and navy under the chief control

of civilians, and by way of compensation hands over the guidance of scientific education to soldiers.

Inorganic Chemical Preparations. By FRANK HALL THORP, Ph.D., Instructor in Industrial Chemistry in the Massachusetts Institute of Technology. Boston (U.S.A.) and London: Ginn and Co. 1896. Pp. 238.

THIS excellent work consists of two main sections:—An introductory or general portion, and a second or experimental part. The former contains general experimental directions, and is very judiciously drawn up. We find a simple precaution against the tiresome phenomenon known as "creeping." Dr. Thorp recommends the edges of the beaker and the evaporating dish to be smeared with a *very thin* layer of paraffin oil or vaseline.

On hydrometers the author speaks very sensibly and emphatically. For liquids heavier than water he pronounces Baumé's hydrometer an utterly unscientific instrument, whose readings bear no very direct relation to true specific gravity. An investigation made a few years ago reveals some thirty-four different scales none of which were correct; yet, in spite of its demonstrably fallacious character, it prevails largely in America and on the European Continent among people who are loud in their condemnation of Britain for her tardiness in adopting the decimal system of weights and measures. With us it is comparatively little used for liquids heavier than water.

An appendix shows the approximate atomic weights and valencies of the elements.

A Short Catechism of Chemistry arranged for Beginners, being an Introduction to the Study of the Science by means of Question and Answer. By ALFRED J. WILCOX. London: Simpkin, Marshall, Hamilton, Kent, and Co. (Ltd.). Middlesbrough: T. Woolston. Entered at Stationers' Hall. Pp. 16.

IF there is still room for another elementary work on chemistry, we may still ask whether the form of question and answer possesses any decided recommendations? The complaint is generally made that the pupil learns without understanding.

In the remarks on the word "Elements" we are told that four of them are gases,—oxygen, hydrogen, nitrogen, and chlorine. Now if the author does not recognise the elementary character of argon and helium, he must surely class fluorine among the gases. It is to be noted that the author, in his Preface, tells us that "his practice of frequently revising with his class the elements of the Science has — and especially before an examination — invariably given excellent results." This we do not in the least dispute; the catechetical system is likely to enable the pupil to answer questions whether he knows or not.

Catalogue of Books by Meyer and Müller, 51, Markgrafen-Strasse, Berlin, W. ("Wegweiser durch die Litteratur der Chemischen Technologie").

A PUBLISHERS' trade catalogue, containing not a few curious works, such as "Margarita Philosophica" (1583), by G. Reisch, "Pandora," the stone of the Muse, by means of which the old philosophi and also Theophrast Paracelsus ennobled the imperfect metals by the power of fire (1588); Helmont (*Opera omnia*, 1648); Guaita, St. de, "Essais de Sciences Maudites." II. Le Serpent de la Genèse. I. "Le Temple de Satan": this book is of as recent a date as 1891!

El Kamlic de Kompozizion ke esperimenta el Agua de "El Salto" durante el Inbierno. By K. NEWMAN. Santiago de Chile. 1896.

THE author's conclusions are:—The water of El Salto undergoes during the winter a chemical and bacteriological

alteration, which causes it to lose its quality and potability. In June and July there existed in the water of El Salto a micro-organism with all the characters of *B. Coli communis*.

CORRESPONDENCE.

THE CYANIDE PROCESS FOR GOLD EXTRACTION.

To the Editor of the Chemical News.

SIR,—Kindly publish for me, as an old subscriber to the CHEMICAL NEWS, the singular and unexpected fact that aqueous solutions of cyanogen do not exert the least solvent action on gold or silver. Of course as the gas decomposes there is a slight solvent action, but even this is far too slow and destructive of the gas to make extraction of gold a commercial success. This must prove to be interesting to cyanide men.

I found this fact while engaged as an expert in the case Government re McDollin and Co., and published it here September 17th last, in a paper to our Philosophical Society.—I am, &c.,

WILLIAM SKEY,
Analyst to the Home Department, N.Z.

The Mines Department, Wellington, N.Z.,
December 5, 1896.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

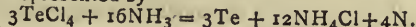
Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 1, January 4, 1897.

Position of the Academy of Sciences. — The following changes have taken place in the membership of the French Academy of Sciences:—Deaths since January 1st, 1896: Resol, Tisserand, Fizeau, Daubrée, Trécul, Reiset, Sappey. There have been elected: Bertrand, Michel Lévy (in the Section of Mineralogy); Muntz (Section of Rural Economy). Among the free Academicians, *i.e.*, those not attached to any especial section, Ronché. The following members have still to be replaced: Resol (Section of Mechanics), Tisserand (Astronomy), Fizeau (General Physics), Trécul (Botany), Sappey (Anatomy and Zoology), and Tchénichef (Foreign Associate). The following correspondents are deceased: Gylden and Gould (Astronomy), Prestwich (Mineralogy), Kékulé (Chemistry), Baron von Müller (Botany), Marquis de Menabrea (Rural Economy). The correspondents elected (Section of Astronomy) are: Gill, *vice* Cayley, deceased; Van de Sande Bakhuyzen, *vice* Newcomb, elected Foreign Associate; Christie, *vice* Hind, deceased. The following correspondents have still to be replaced: Gylden and Gould, deceased (Astronomy); Kékulé, deceased (Chemistry); Prestwich, deceased (Mineralogy); Baron Müller, deceased (Rural Economy); Marquis de Menabrea, deceased (Rural Economy); Lovén, deceased (Anatomy and Zoology).

Effects of the Combined Variation of the Two Factors of the Expenditure of Muscular Energy on the Value of the Respiratory Exchanges.—A, Chauveau, with the assistance of J. Tissot.—This paper is too little and too doubtfully chemical in its character to merit insertion or abstraction in the CHEMICAL NEWS.

Action exerted upon the Alkaline Haloid Salts by the Bases which they contain.—A. Ditte.—The alkaline bases exert upon the alkaline haloid salts a precipitating action analogous to that produced by the corresponding acids.

Action of Ammonia upon Tellurium Bichloride.—René Metzner.—Ammonia acts upon tellurium bichloride in a manner which differs according to the temperature applied. At 200° to 250° the reaction, which is very slow, is represented by—



At 0° the ammonia combines with tellurium chloride. At a lower temperature we may obtain combination of tellurium and nitrogen. When the operation succeeds the apparatus contains, after the washing is completed, a substance of a fine lemon-yellow colour, of the composition TeN. This nitride is friable and amorphous. It detonates with extreme violence if struck, producing a black vapour of tellurium in an impalpable powder. Tellurium nitride is not attacked either by water or dilute nitric acid. In contact with potassia it gives off all its nitrogen in the state of ammonia.

The Absorption of Hydrogen Sulphide by Liquid Sulphur.—H. Bélabon.—Liquid sulphur maintained at a temperature above 170°, in presence of sulphuretted hydrogen gas, absorbs a notable quantity of this gas. The quantity of gas absorbed is so much the greater as the temperature is higher, the pressure remaining the same. In all cases the gas escapes at the moment of the solidification of the sulphur; the gaseous liberation is a consequence of solidification. Pure hydrogen is not absorbed by liquid sulphur.

Production of Vanilline by means of Vanillo-Carbonic Acid.—Ch. Gassmann.—The author boils 1 part of this acid with 2 parts of aniline until the escape of carbonic acid has ceased. There is formed a substituted benzylidene-aniline, which is separated from excess of aniline by means of a current of steam. The vanilline-aniline is finally split up by a brief ebullition with dilute sulphuric acid at 50 per cent. The vanilline formed is isolated by extraction with ether, from which it easily crystallises.

The Transformation of Eugenol into Isoeugenol.—Ch. Gessmann.—Not adapted for useful abstraction.

A New Method for Determining Sulphur in Iron.—W. Schulte.—In order to evade the disadvantages of the bromine method and of working with barium sulphate the author dissolves the iron in dilute hydrochloric acid, conducts the gases evolved through a solution of cadmium acetate acidulated with acetic acid, and transforms the resulting cadmium sulphide in order to bring it into a state capable of easy determination into copper sulphide by the addition of an acid solution of copper sulphate. The copper sulphide is ignited and weighed as copper oxide. One atom of sulphur yields exactly 1 mol. of copper oxide; $31.98\text{ S} = 79.14\text{ CuO}$. It is not admissible to pass the gases evolved into the copper solution, since phosphorus and arsenic occasion separations. We prepare in the first place three solutions. I. 25 grms. cadmium acetate (or 5 grms. cadmium acetate + 20 grms. zinc acetate) and 200 c.c. glacial acetic acid per litre; II. dilute hydrochloric acid (1+2); III. 80 grms. copper sulphate and 175 c.c. concentrated sulphuric acid per litre. The apparatus consists of a boiling flask, into which are introduced 10 grms. comminuted iron, a funnel tube with a cock for the introduction of 200 c.c. hydrochloric acid, a bent glass tube as a reflux refrigerator, and a receiver with an appendix and a second safety receiver. Into the receiver there are introduced 40 to 50 c.c. of the cadmium solution. The development of gases is effected in the cold, heat is then applied with a Bunsen burner so as to complete the solution of 10 grms. iron in ninety minutes. The cadmium compound in the receiver is then transformed with 6–7 c.c. of the copper solution, the copper sulphide is then filtered off and ignited. If it is intended in this manner to determine zinc and manganese sulphides in iron sulphide, the portion operated upon must not exceed 0.15 gm. For 10 grms. iron the entire process requires two-and-a-half hours.—*Stahl und Eisen*

MISCELLANEOUS.

Mica.—Messrs. Wiggins and Sons, Mica Merchants, have, after rebuilding, returned to their old address, 102 and 103, Minorities, both of which buildings are entirely devoted to their business.

Royal Institution.—On Saturday, Jan. 23, Mr. Carl Armbruster will deliver the first of three lectures on "Neglected Italian and French Composers" (with numerous vocal illustrations). The Friday Evening Meetings of the members will commence on Jan. 22, when Prof. Dewar will deliver a lecture on "Properties of Liquid Oxygen." Prof. J. C. Bose, Professor of Presidency College, Calcutta, will deliver his discourse on "The Polarisation of the Electric Ray" on Friday evening, Jan. 29th, and not on Feb. 5th as previously announced. The discourse on this night will be delivered by the Bishop of London, who will take as his subject "The Picturesque in History."

Sensitive Litmus-Paper.—Ronde.—The strong alkaline cubes occurring in commerce are covered with twelve to fifteen times their quantity of water, and allowed to stand for one day. The deep blue mixture is then treated with sulphuric acid until it becomes a light red, and heated on a steam-bath for fifteen minutes. To the liquid, which generally turns blue again, dilute sulphuric acid is added until the filter-paper becomes of a reddish-violet on immersion. When cold it is strained through a cloth, and the liquid is so adjusted, by the addition of drops of dilute sulphuric acid or traces of powdered litmus, that pieces of filter-paper, if immersed and quickly dried, take the desired red or blue tint. This method readily yields papers of a sensitiveness $\tau = 150,000$.—*Pharm. Zeitung and Chem. Zeitung.*

Royal Academy of Sciences of Turin.—From a courteous communication from the Academy we learn that from January 1st, 1897, to the end of December, 1898, this prize will be awarded to any scientific author or inventor, of whatever nationality, who, during the years 1895—98, shall, in the judgment of the Royal Academy of Sciences of Turin, have made the most important discovery or published the most valuable work on physical and experimental science. The prize (deducting income-tax) will be 9600 francs. Candidates must send in this work (*in print*) to the President of the Academy within the stated time. MSS. will be disregarded. Unsuccessful work will not be returned.—Signed, G. CORB (President); E. D. ONDICI (Secretary of the Commission); Turin, January 1st, 1897.

MEETINGS FOR THE WEEK.

MONDAY, 25th.—Society of Arts, 8. (Cantor Lectures). "Material and Design in Pottery," by Wm. Burton, F.C.S.
TUESDAY, 26th.—Royal Institution, 3. "Animal Electricity," by Prof. A. D. Waller, F.R.S.
— Society of Arts, 8. "The Artistic Treatment of Heraldry," by W. H. St. John Hope, M.A.
WEDNESDAY, 27th.—Society of Arts, 8. "Voice Production," by William Nicholl.
THURSDAY, 28th.—Royal Institution, 3. "Some Secrets of Crystals," by Prof. H. A. Miers, F.R.S.
— Society of Arts, 4.30 (at the Imperial Institute). "The Moral Advancement of the Peoples of India during the Reign of Queen Victoria," by William Lee-Warner, M.A., C.S.I.
— Society of Arts, 8. "The Mechanical Production of Cold," by Prof. James A. Ewing, M.A., F.R.S.
FRIDAY, 29th.—Royal Institution, 9. "The Polarisation of the Electric Ray," by Professor Jagadis Chunder Bose, M.A., D.Sc.
SATURDAY, 30th.—Royal Institution, 9. "Neglected Italian and French Composers," by Carl Armbruster.

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1940.

THE UNITY OF THE ATOMIC WEIGHTS.

By KARL SEUBERT.

IN a Report by F. W. Küster, on the Progress of Physical Chemistry in the year 1895, which lately appeared in the *Zeit. Anorg. Chem.*, it is mentioned that "all the more recent determinations of the atomic weight of hydrogen show in accordance that the relation O : H lies between 16:1.007 and 16:1.008 (or, in the old style, H : O between 1:15.87 and 1:15.88)." We read further that this result, which is not novel, will scarcely prevent the value H : O = 1:15.96 from being still quietly employed. I cannot share the apprehension of my colleague, Küster. I have at once, after Morley's great work had become known to me in its details, recognised the inadmissibility of the value 15.96, and have expressed this conviction on the next opportunity. The remaining adherents of the "old style" may probably think with me, since what formerly led us to the assumption of the value O = 15.16 is no longer valid, this number no longer presenting the result of the most trustworthy experimental determinations of the atomic weight of oxygen in reference to the atomic weight of hydrogen taken as unity. This result has seemed for some time probable, but the question was too much contested to admit of a full decision, and a certain reserve appeared to me the more imperative as now and then determinations spoke in favour of the older values 15.96 and 16. At present we may, however, admit that Morley's relation, O : H = 15.879:1, approaches so closely to the truth that it will not in future undergo any modification of importance. The uncertainty, in all probability, amounts to not more than a unit in the second decimal place, or about 0.06 per cent of entire value. Hence the atomic weight of oxygen now ranks among those most accurately determined, and its possible error becomes perceptible only in the higher values of atomic weights, and even there in decimal places which in any case must be held doubtful.

There is near at hand an expedient to avoid the unpleasantness involved in the uncertainty and the change in the atomic weight of oxygen by fixing this value arbitrarily, and thus rendering it independent of the issue of stöchiometrical determinations. This expedient must, however, be used with the utmost possible limitation. I should not delay for a moment to vote to-day for O = 16, if the result of the most recent investigations on the atomic weight of oxygen evinced considerable deviations and uncertainties which rendered a selection among the different values impossible; but, on the contrary, these researches, almost without exception, lead to a value derived from an experimental investigation conducted on a large scale and in an unexceptional manner, and invest it with a degree of probability which attaches to our best determinations of atomic weights. If, therefore, a number must be established for the atomic weight of oxygen, that value has the first claim which—according to our present experience—gives our atomic weights a rational unity, the atomic weight of hydrogen.

An advantage which has been put forward in favour of the assumption O = 16 is, that then a number of atomic weights can be reproduced with sufficient accuracy by whole numbers; but this plea is not sufficient to turn the scale in its favour. The new atomic weights are certainly less convenient in this respect.

They show that Prout's hypothesis, both in its original form and in the modification proposed by Dumas, is not refuted by experiment. The atomic weights of the ele-

ments—if we disregard desultory exceptions—are neither multiples of the atomic weights of hydrogen nor of 0.5 nor of 0.25, therewith the conclusions attached to this supposed regularity become baseless.

		O = 16.	H = 1.
Aluminium	Al	27.11	26.90
Antimony	Sb	119.9	119.0
Arsenic	As	75.1	74.5
Barium	Ba	137.43	136.40
Beryllium	Be	9.05	8.98
Bismuth	Bi	208.9	207.3
Boron	B	10.94	10.85
Bromine	Br	79.96	79.35
Cadmium	Cd	112.0	111.1
Carbon	C	12.00	11.91
Calcium	Ca	40.01	39.71
Cæsium	Cs	133.0	132.0
Cerium	Ce	140.3	139.2
Chlorine	Cl	35.46	35.19
Chromium	Cr	52.14	51.74
Cobalt	Co	59.6	59.1
Copper	Cu	63.60	63.12
Didymium	Di	142.5	141.4
Erbium	E	166.4	165.1
Fluorine	F	19.10	18.96
Gallium	Ga	70.1	69.6
Germanium	Ge	72.5	71.9
Gold	Au	197.2	195.7
Hydrogen	H	1.008	1.00
Indium	In	114.0	113.1
Iridium	Ir	193.0	191.5
Iron	Fe	56.02	55.60
Iodine	I	126.86	125.90
Lanthanum	La	138.3	137.3
Lead	Pb	206.91	205.35
Lithium	Li	7.03	6.97
Magnesium	Mg	24.26	24.18
Manganese	Mn	54.94	54.52
Mercury	Hg	200.3	198.8
Molybdenum	Mo	96.03	95.31
Nickel	Ni	58.9	58.4
Niobium	Nb	93.9	93.2
Nitrogen	N	14.04	13.94
Osmium	Os	190.8	189.3
Oxygen	O	16.00	15.88
Palladium	Pd	106.3	105.0
Phosphorus	P	31.03	30.80
Platinum	Pt	194.8	193.3
Potassium	K	39.12	38.83
Rhodium	Rh	103.0	102.2
Rubidium	Rb	85.4	84.8
Ruthenium	Ru	101.7	100.9
Scandium	Sc	44.05	43.75
Selenium	Se	79.07	78.47
Silver	Ag	107.92	107.11
Silicon	Si	28.38	28.16
Sodium	Na	23.05	22.88
Strontium	Sr	87.62	86.96
Sulphur	S	32.06	31.82
Tantalum	Ta	182.5	181.1
Tellurium	Te	127.5	126.6
Thallium	Tl	204.2	202.7
Thorium	Th	232.5	230.7
Tin	Sn	119.10	118.20
Titanium	Ti	48.1	47.8
Tungsten	W	184.1	182.7
Uranium	U	239.4	237.6
Vanadium	V	51.2	50.8
Ytterbium	Yb	173.0	171.7
Yttrium	Y	88.9	88.3
Zinc	Zn	65.41	64.91
Zirconium	Zr	90.6	89.9

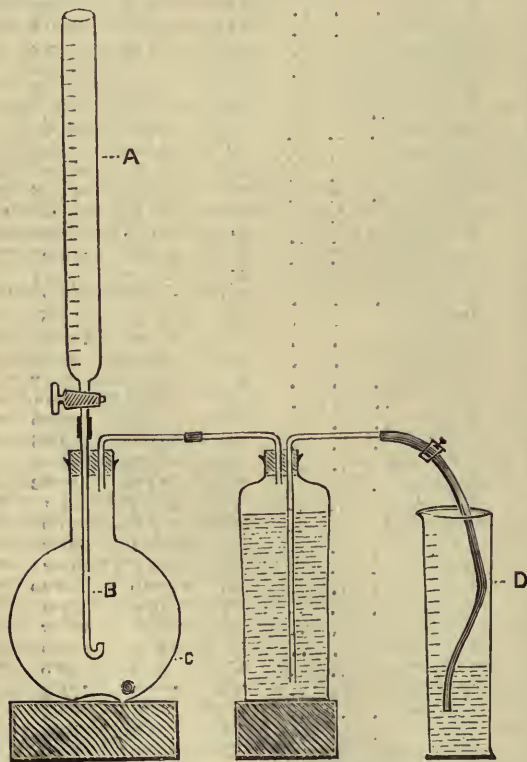
If Küster further remarks that "the selection of hydrogen as the unit of the atomic weights has been often branded as illogical, and practically not to be upheld," I

cannot share his opinion. It seems to me most natural and logical to refer the atomic weights to the smallest of all—that of hydrogen—as the unit. If this is not feasible from practical reasons, another unit must be selected out of the series of the atomic weights, as it has been done in the case of specific gravities. The unquestionably inconvenient numbers which result on referring to $O = 1$ have led to the expedient of taking the atomic weight of oxygen = 16.00, and thus creating a unit identical with no known atomic weight, though it approximates to that of hydrogen. *Per se*, this choice is illogical, and the number 16 only obtains meaning with reference to hydrogen. It represents makeshift, founded on the idea that the reference to hydrogen as unit would be in principle more accurate. Practically we have obtained such a relation by assuming $O = 15.879$, for which we may more briefly substitute 15.88, for in all probability future researches will modify this value so little that the influence of such a correction will be felt in most atomic weights only in places which are already uncertain. It is very questionable whether the advantages held out for the calculation $O = 16.00$ will counterbalance the advantage of a really rational, logical unit.—*Zeit. Anorg. Chem.*

NOTE ON THE DETERMINATION OF EQUIVALENT OF SODIUM.

By WM. FRENCH.

FOR the determination of the equivalent of sodium by junior students I have adopted the following method, which I find has many advantages over the sodium amalgam method mentioned in most practical text-books.



I have not seen a notice of this modification in any of the numerous works on practical chemistry, and assume, therefore, that it is certainly not generally adopted. The

accompanying sketch will explain the experimental part of the process. The tube B is completely filled by the stoppered burette, A, with dilute alcohol (of such strength that sodium dissolves in it at a gentle rate). A piece of sodium is quickly weighed and placed in the small dry flask, C, which is fitted on to the cork connected to a water syphon aspirator in the usual way. Dilute alcohol is then run on to the sodium from the burette, and the volume of water displaced after due cooling and levelling in the measuring cylinder, D, minus the volume of alcohol run in from the burette, gives the volume of hydrogen evolved, from which the equivalent of sodium can be calculated.

Grammar School, Bury.

THE TOXICOLOGICAL BEHAVIOUR OF PICRIC ACID AND ITS SALTS, AND OF CERTAIN KINDRED SUBSTANCES.

By Dr. TH. BOKORNY.

FREE picric acid is a powerful poison for algæ; in a 0.5 per cent solution they dried within fifteen minutes; in 0.1 and 0.05 per cent solutions within twenty-four hours. Many fungi are not quite so sensitive to picric acid.

Free picric acid is no powerful poison for fungi. With algæ a concentration of 0.05 per cent is sufficient for poisoning, whilst fungi require one of 0.1 per cent. Ammonium picrate is for low organisms a more powerful poison than potassium picrate.

In a solution of orthobenzoic acid of 0.2 to 0.1 and 0.05 per cent all life of algæ and low animals was extinguished in five hours.

Potassium nitrobenzoate in a 0.2 per cent solution destroys all animal and vegetable life within six hours.

According to Weyl, Martius yellow or dinitro- α -naphthol is a strong poison for dogs, the fatal dose being 0.3 gm. per kilo. living weight. Naphthol yellow S, a sulpho-acid of dinitro- α -naphthol, is harmless. A comparison between ortho- and para-nitrophenol shows that the para-compound is somewhat more poisonous to algæ and infusoria than the ortho-compound. It would be interesting to carry out a comparison between para- and ortho-compounds in an extensive series of organic compounds.—*Chemiker Zeitung*.

DETERMINATION OF BISMUTH.

By W. MUTHMANN and F. MAWROW.

THE authors have shown some time ago that hypophosphorous acid is suitable for the determination of copper, and especially for its separation from cadmium and zinc. Experiments with bismuth have now shown that with suitable precautions it may be completely precipitated as metal by the reagent above named. Hence there results a method of separation and determination which in many cases surpasses in accuracy and expedition the methods hitherto known. We shall briefly describe it.

The solution of the bismuth salt, not too strongly acid, is mixed with an excess of hypophosphorous acid, and heated on the water-bath until the supernatant liquid has become perfectly clear, and a further addition of the reagent heated to ebullition produces no further colouration. The metal separates out in the form of a reddish-grey spongy mass, which can be easily filtered and washed. It is collected upon a weighed filter or in a Gooch crucible, washed with boiling water and then with absolute alcohol, and dried at 105°. The original solution is best used in a state of moderate concentration, and a few c.c. of hypophosphorous acid are forthwith added.

Our experiments have been carried out with bismuth oxychloride washed up in a little water and dissolved by the addition of a few drops of hydrochloric acid. The liquid after filtration was tested each time for bismuth by the introduction of hydrogen sulphide. There only appeared a faint brown colouration if heat had not been applied for a sufficient time. In four experiments there were used respectively—

BiOCl.	Obtained.	Bi per cent.
0.158 grm.	0.0926 grm.	79.96
0.1559 "	0.1250 "	80.17
0.1201 "	0.0963 "	80.18
0.1068 "	0.0857 "	80.24

The average percentage being 80.13, and the calculated result 80.15.

The method is doubtless applicable for the separation of bismuth from metals which are not precipitated from their solutions by hypophorous acid, especially from zinc and cadmium.—*Zeit. Anorg. Chemie*, xiii., p. 207.

SÄRNSTRÖM'S METHOD OF DETERMINING MANGANESE IN IRON ORES.

By C. T. MIXER and H. W. DUBOIS.

ABOUT a year ago we had occasion to use a volumetric method which would allow the determination of manganese in iron ores ranging in amounts up to 15 per cent, and give results in half-an-hour which would check with gravimetric determinations within two-tenths per cent for ores as high as 15 per cent, and within a few hundredths of a per cent for ores under 1 per cent.

We found in use in a neighbouring laboratory a method which was generally known as the "Swedish Method." This method was found to fulfil the above conditions, and seems to have sufficient merit to be more widely known.

The first suggestion upon which the method is based was made by Guyard,* although his method of operating it did not give very satisfactory results.

The first description of the method in its present practical form was made by C. G. Särnström, in the *Jernkontorets Annaler* (Sweden), 1881, p. 401.†

The principle upon which this method depends is the reaction which takes place when a manganese compound higher in oxygen than the manganous state, is dissolved in hydrochloric acid, forming a higher chloride, which is readily shown by the dark coloured solution. When this solution is boiled, it rapidly decolourises, being completely converted into manganous chloride, not easily oxidised by the air while in the acid solution.

In neutral or alkaline solutions the manganous compound has a slight tendency to oxidise in contact with the air, but we have never detected any appreciable oxidation under the conditions which we follow.

The separation of the iron and the manganese is effected in such a way that the iron is precipitated as hydroxide, and the manganese left in the manganous condition in solution. Sodium carbonate is used to precipitate the iron as hydroxide, and no trouble is experienced in the precipitation of the manganese as a carbonate, provided that only a very slight excess is employed beyond that necessary to completely precipitate the iron. It is advisable to add the sodium carbonate in the form of a solu-

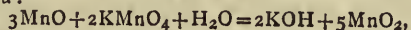
tion, towards the completion of the precipitation of the iron, to avoid such an excess.

Särnström employs sodium bicarbonate, which has the advantage that a greater amount of carbon dioxide is generated, preventing subsequent oxidation of the manganous salt by the oxygen of the air. Manganese bicarbonate is formed, which is readily soluble in the solution containing carbon dioxide.

It is always desirable to test the sodium carbonate or bicarbonate for organic matter.

The results given below show the necessity of avoiding an excess of sodium carbonate (the same is true of the bicarbonate) in the precipitation of the iron. Aliquot portions of a solution of manganese containing 3.14 per cent manganese gravimetrically determined, gave only 2.61 per cent when treated with such an excess. Another ore giving 8.69 per cent under the proper conditions of this method, when treated with an excess of sodium carbonate gave 5.38 per cent.

After the precipitation of the iron in the hot solution, the manganese being in the manganous state, is oxidised by potassium permanganate, according to the following formula:



which is the same reaction which takes place in Volhard's method. As the titration takes place directly, without filtering, the precipitate of ferric hydroxide is an advantage, especially in low manganese ores, as it serves to collect the fine precipitate of manganese dioxide and causes it to settle more rapidly.

In ores very low in iron it is desirable to add ferric chloride in order to obtain the requisite amount of the iron precipitate.

The Method.—Weigh half a grm. ore into a No. 0 beaker, add 15 c.c. of hydrochloric acid, 1.1 sp. gr., and boil until the residue is clear. If necessary fuse the residue with sodium carbonate. Add a few drops of nitric acid to oxidise any ferrous iron or organic matter. In magnetic ores more of course will be necessary. It is well to test for ferrous iron. Evaporate a short time to expel any nitrous acid that may have been formed. It is advisable to have a good amount of free hydrochloric acid present to generate carbon dioxide in the precipitation with sodium carbonate. The solution is then washed into a No. 3 beaker or a flask, which is then filled about two-thirds full with boiling distilled water, and solid sodium carbonate or bicarbonate added until the iron is completely precipitated, which is readily indicated by the characteristic spongy appearance of the precipitated ferric hydroxide. A solution of the salt is preferable for the final precipitation in order to avoid an excess.

The solution should be about 80° C. when it is titrated* with potassium permanganate directly, without filtering, and with intervals of vigorous stirring and settling of the iron and manganese precipitates, until the supernatant liquid shows a permanent faint pink colour. The first appearance of the pink colour must not be taken as an indication that the oxidation is complete, as gentle heating and vigorous stirring will allow more potassium permanganate to be added before the permanent pink appears.†

Multiplying the burette reading by two represents the equivalent for one grm., and this multiplied by the permanganate value in manganese, which is the iron value multiplied by 0.2946, gives the percentage of manganese.

In case of over-titration, it is practicable to titrate back with a carefully standardised solution of manganous chloride, which is prepared by evaporating 15 c.c. potassium permanganate down to 3 or 4 c.c., adding a few drops of hydrochloric acid and boiling as long as chlorine comes off. The solution should be neutralised with sodium carbonate, and diluted to 10 c.c., when 1 c.c. is equal to 1 c.c. of potassium permanganate.

* Which should be done immediately after the neutralisation, in order to avoid any opportunity for oxidation.

† This is a very important point not only in relation to this method, but in all methods where potassium permanganate is used.

* Guyard, *CHEM. NEWS*, viii., 292; the following references relate to the subsequent modifications of the method; Habich, *Ztschr. Anal. Chem.*, iii., 474; Winkler, *Ztschr. Anal. Chem.*, iii., 423; Morawski and Singl, *Journ. Prakt. Chem.* (N.F.), xviii., 96; Volhard, *Ann. Chem.* (Liebig), cxviii., 318.

† Also published in Berg und Hüttenm., *Zeitung*, xl, 425. A review of the original article appears in the *Ztschr. Anal. Chem.*, xxii., 84. We are indebted to Mr. Hugo Carlsson, Chief Chemist of the Johnson Works of Lorain, Ohio, for calling our attention to Särnström's original publication and for furnishing us a translation of the same.

Särnström states that the method is reliable for high manganese ores and ferro-manganese "where it is not necessary to determine the manganese closer than a few tenths of 1 per cent."

Our experience does not confirm this. The results average from 1 to 2 per cent too low, so that we do not consider the method at all reliable for high percentages of manganese.

The following are some results which show this:—

	Särnström. Per cent.	Gravimetric (Ford's) Per cent.
Illinois ore	52.06 (1)	52.98
"	51.91 (2)	—
"	51.40 (3)	—
"	51.78 (4)	—
"	51.37 (5)	—
"	51.37 (6)	—
No. 595	42.07	—
"	42.35	44.3
"	42.90	—
"	42.60	—

In analyses from No. 1 to No. 4 sodium carbonate was used for the precipitation. To determine whether the employment of sodium bicarbonate would be advantageous, No. 6 was so treated, while at the same time No. 5 was precipitated with sodium carbonate, yielding the same result. We have tried sodium bicarbonate with low manganese ores, but have never noticed any practical advantages, while theoretically, as we have pointed out above, there should be an advantage in the employment of sodium bicarbonate.

This discrepancy with high percentages of manganese may possibly be accounted for by the fact that the large precipitate of manganese dioxide may act in a purely mechanical way in protecting the final amounts of the manganous chloride from being fully oxidised to dioxide by the potassium permanganate. It is to be noted in this connection that Volhard's method does not generally give reliable results with such high percentages of manganese.

The following are some results obtained by this and other methods:—

	Särnström.	Volhard.	Gravimetric.
A. Magnetic ..	0.07	0.10	0.07
Specular ..	—	0.08*	—
B. Mixture of blue granu- lar and red hematite	0.30 0.32 0.28	0.31 0.29 —	0.29 0.30 —
C. Limonite ..	1.03	1.02	1.09
" ..	1.05	1.05	—
D. Silicious ore	2.98	3.08	2.93
" ..	3.07	3.07	—
Cary Empire ..	3.93	T. V. Church	3.94
" ..	3.88	Illinois Steel Co.	
Dexter No. 2 ..	6.04 6.02 6.01	— 6.02 —	6.01 — —
Davis ore ..	8.78	8.62	8.86
Newark ore ..	1.48	A. G. McKenna,	(Ford's) 1.50
No. 57 ..	5.39	Duquesne Steel Works	
No. 218 ..	5.59	5.59	—

In the determination of small amounts of manganese this method presents an advantage over Volhard's method in giving a more distinct end reaction.

The method can be used for iron and steel determinations if the usual precautions are taken to oxidise the carbon. But it is not so well adapted to these on account of the impracticability of taking large amounts for analysis. —*Journal of the American Chemical Society*, xviii., April, 1896.

* This was so low as to necessitate filtering through asbestos in order to see end reaction by Volhard's method.

METAL SEPARATIONS BY MEANS OF HYDROCHLORIC ACID GAS.*

By J. BIRD MOYER.

Introduction.

THE action of gaseous haloid acids upon metallic oxides and their salts is a field of investigation which, though not of recent origin, has been but lately developed. It was Debray (*Comp. Rend.*, xlv., 1098, and *Ann. Chem.* (Liebig), cviii., 250) who first called attention to the volatility of molybdic acid in a stream of hydrochloric acid gas, with the formation of $\text{MoO}(\text{OH})_2\text{Cl}_2$.

E. Péchard (*Comp. Rend.*, cxiv., 173) applied this and showed that molybdic acid was completely eliminated and separated from tungstic acid by its volatility in a current of hydrochloric acid. Since that time nothing further has been done with single haloid acids, in gas form, until quite recently. Compounds have been decomposed, salts volatilised, and separations made, by means of other gases and mixtures, which may be as effective as hydrochloric acid, but are not devoid of trouble nor nearly so neat.

Smith and Oberholtzer (*Journ. Amer. Chem. Soc.*, xv., 1), repeated and confirmed Péchard's work in regard to the separation of molybdic acid from tungstic acid, and in addition showed that gaseous hydrobromic, hydriodic, and hydrofluoric acids acted similarly. Later, Smith and Maas (*Ztschr. Anorg. Chem.*, v., 280) made use of the volatilisation of molybdic acid for a close atomic mass determination of molybdenum.

Smith and Hibbs (*Journ. Amer. Chem. Soc.*, xvi., 578) showed that vanadium behaved like molybdenum. Hydrochloric acid gas completely eliminates vanadic acid from sodium vanadate. A little later they investigated the action of hydrochloric acid upon the members of Group V. of the periodic system (*Ibid.*, xvii., 682).

The sodium salts of nitric, pyrophosphoric, pyroarsenic, and pyroantimonic acids were used. They found nitrogen, arsenic, and antimony to be volatile in gaseous hydrochloric acid, and made it the basis of a separation of phosphoric acid from nitric acid. Lead arsenate changed completely to chloride, the arsenic being volatilised, thus affording a good quick separation. Smith and Meyer (*Ibid.*, xvii., 735) tried the action of all the haloid acids upon the elements of Group V. of the periodic system. They worked with sodium salts and observed:—I. That nitrogen was expelled completely by all the haloid acids. II. That phosphoric acid was not acted upon. III. That arsenic acid was fully expelled by hydrochloric, hydrobromic, and hydriodic acids, but only partially by hydrofluoric acid. IV. That antimony was completely volatilised by hydrochloric acid. There was no work done on bismuth. V. Vanadium went over completely in hydrochloric acid, but only partially in hydrobromic and hydrofluoric acids. VI. Columbium forms volatile products with hydrochloric and hydrobromic acids. No knowledge of didymium was obtained. VII. Tantalum is only slightly volatile in hydrochloric acid.

P. Jannasch and F. Schmidt (*Ztschr. Anorg. Chem.*, ix., 274) repeated some of the work of Smith and Hibbs, in which they confirmed the separation of arsenic from lead. They anticipated a slight portion of my work, and in addition separated arsenic acid from iron, tin from lead, tin from copper, and tin from iron, in a stream of hydrochloric acid gas.

The position of bismuth in the periodic system makes it natural to suppose that it too will be volatile in hydrochloric acid gas. This I have shown to be true, and was thus enabled to separate it from lead and copper. The action of hydrobromic acid on bismuth trioxide was also tried; it formed the bromide and then volatilised. It

* From author's thesis presented to the Faculty of the University of Pennsylvania for the degree of Ph.D., 1896. From the *Journ. Amer. Chem. Soc.*, xviii., December, 1896.

requires a higher temperature and longer action than with hydrochloric acid. Because of lack of time, I have been compelled to abandon the experiments instituted with a view of affecting separations, in atmospheres of hydrobromic acid and hydriodic acid gas, and have confined my labours to hydrochloric acid gas.

Method of Work.

The hydrochloric acid gas was generated by dropping concentrated sulphuric acid from a separatory funnel upon concentrated hydrochloric acid contained in a three litre flask. The gas evolved at the ordinary temperature was dried by passing it through two sulphuric acid drying bottles and then through a calcium chloride tower, when it was considered sufficiently dry for the purpose. The substance to be acted upon was weighed out in a porcelain boat, and the latter was placed in a combustion tube of hard glass.

The tube had previously been rinsed with alcohol and then with ether to remove all moisture. The ether was removed by drawing a current of dry air through the tube. This tube was connected to a two-necked bulb receiver containing about 300 c.c. of distilled water. When working with arsenic 10 c.c. of nitric acid were added. The connecting tube from the combustion tube to the bulb receiver was made to enter the receiver and dip below the surface of the water, thus catching all volatile products, as well as taking up the hydrochloric acid gas. To insure safety from the loss of volatile products, a small flask containing water was attached to the bulb receiver. The apparatus was controlled at both ends by stop-cocks. This is necessary to prevent backward suction on disconnecting the apparatus. After the reaction was completed the boat was removed to a sulphuric acid desiccator from which the air could be exhausted. In general, the procedure was similar to that employed by Hibbs (Thesis, 1896).

I.—Behaviour of Antimony Trioxide.

Antimony oxide, labelled chemically pure, was dissolved in hydrochloric acid and precipitated with a large amount of water. After washing by decantation it was re-dissolved and re-precipitated. This procedure was repeated several times, when it was precipitated by ammonium carbonate, washed, and ignited. The pure oxide obtained in this manner was subjected to the action of hydrochloric acid gas, and it was found to volatilise completely. In each trial a one-tenth grm. of the oxide was acted upon. The temperature varied between 150° and 190° C. It was determined in the following way:—The combustion tube was slipped through two holes made in the sides of a copper drying oven.

A very slow current of gas was used as the antimony seemed to volatilise more readily and completely if the current was slow and the heat gentle. This I attribute, on reflection, to the fact that I ignited the oxide too strongly (to a red heat) in its preparation. It dissolved with difficulty in concentrating hydrochloric acid. Lack of time prevented the repetition of this experiment and the separation of antimony from lead and copper, in which this substance was used. About eight hours was the time required for the volatilisation; very probably a shorter time would be required if the oxide had been obtained by gentle ignition.

II.—Behaviour of Lead Oxide.

Pure lead oxide was obtained from re-crystallised nitrate by careful ignition. This oxide changed completely into chloride at the ordinary temperature, and it was only necessary to apply a gentle heat to complete the change and entirely remove the water formed. No volatilisation was noticed until a temperature of 225° was reached; at this point the lead chloride slightly volatilised.

I think it possible to estimate lead as chloride, if the temperature is kept under 200°. A weighed amount of lead oxide was acted upon by hydrochloric acid gas in the

cold for two hours, and then heated sufficiently to remove all the water formed.

The boat was cooled in the gas, and then placed in a sulphuric acid desiccator and allowed to stand one-half hour. It was then weighed.

Experiments.

	Lead oxide taken.	Lead chloride obtained.	Lead chloride required.	Difference.
	Grm.	Grm.	Grm.	Grm.
Experiment I.	0.1017	0.1267	0.1267	0.0000
" II.	0.1015	0.1258	0.1265	-0.0007
" III.	0.1169	0.1454	0.1447	+0.0007

The lead chloride dissolved in hot water without residue.

III.—The Separation of Antimony from Lead.

The oxides were carefully weighed and thoroughly mixed in a porcelain boat. Hydrochloric acid gas was passed over them in the cold, until the lead oxide had been entirely changed to the chloride. It was then heated with the smallest flame obtainable from a fish-tail burner, placed about two inches below the tube.

	Antimony trichloride taken.	Lead chloride taken.	Lead chloride obtained.	Lead chloride required.
	Grm.	Grm.	Grm.	Grm.
Experiment I.	0.1015	0.1189	0.1470	0.1482
" II.	0.1090	0.1021	0.1266	0.1272
" III.	0.1350	0.0852	0.1057	0.1062
" IV.	0.1250	0.1671	0.2069	0.2083

The time required was seven hours. The lead chloride was immediately weighed. It dissolved completely in hot water, and this solution was tested by means of Marsh's apparatus for antimony, without finding the latter present. Experiment II. was slightly varied by first moistening the oxides with a drop of hydrochloric acid.

IV.—Behaviour of Bismuth Oxide.

Bismuth nitrate, as pure as could be obtained, was dissolved in nitric acid and then thrown down with a large quantity of water. The precipitate was carefully washed by decantation. This operation was repeated several times.

It was then dissolved in acidulated water and precipitated with ammonium hydroxide and ammonium carbonate. This, on ignition, gave pure oxide, which, heated in a stream of hydrochloric acid gas, completely volatilised as chloride. Here the same treatment is necessary as obtained for antimony. A slow current of gas and a low heat were best adapted for the volatilisation (a temperature of 130°, or roughly, the heat afforded by a fish-tail burner placed two inches below the combustion tube, with a flame an eighth of an inch high). The bismuth chloride sublimed nicely, forming a white crystalline mass beyond the boat, which could be readily driven along by a gentle heat.

V.—The Separation of Bismuth from Lead.

The same material was used as in the preceding experiments. The weighed oxides were thoroughly mixed in a porcelain boat. Usually the gas was allowed to act in the cold for an hour, which changed the oxides completely to chlorides.

The same conditions prevailed as under bismuth oxide alone. If an attempt was made to hasten the reaction by heating higher than 180°, a little lead would volatilise. This sublimate, slightly yellow in colour, would appear directly over the boat and could not be driven along the tube like bismuth; hence it was readily detected.

The separation of bismuth from lead requires much care, as it is not as sharp as could be desired. It is also difficult to tell exactly when the last traces of bismuth have been driven out of the boat, as there was no colour change to indicate it, both metals forming white chlorides. The separation is complete in from six to seven hours. At the end of the separation the position of the boat was

changed and the action continued; if no further sublimation occurred it was cooled and removed to a desiccator. The weight was taken after standing one-half hour over sulphuric acid. With care bismuth can be separated from lead in this manner.

	Lead. oxide taken.	Bismuth trioxide taken.	Lead chloride obtained.	Lead chloride required.	Difference.
	Grm.	Grm.	Grm.	Grm.	Grm.
Experiment I.	0.01014	0.02020	0.1261	0.1264	-0.0003
" II.	0.01006	0.0642	0.1252	0.1254	-0.0002
" III.	0.01038	0.1003	0.1294	0.1302	-0.0008
" IV.	0.01412	0.1260	0.1759	0.1759	0.0000

The chloride of lead dissolved completely in hot water. It showed no bismuth. The sublimate contained no lead.

(To be continued).

THE DETERMINATION OF ATOMIC MASSES OF SILVER, MERCURY, AND CADMIUM, BY THE ELECTROLYTIC METHOD.*

By WILLETT LEPLEY HARDIN.

(Continued from p. 41).

SECOND SERIES.

Experiments on Silver Acetate.

THE fact that silver forms well-crystallised salts with a number of organic acids makes the comparison of the atomic mass of silver with the combined atomic masses of carbon, hydrogen, and oxygen, a matter of no great difficulty. From certain preliminary experiments the acetate of silver seemed to fulfil the conditions necessary for accurate determinations.

Preparation of Silver Acetate.

The purest commercial sodium acetate was dissolved in water, the solution filtered and re-crystallised. After three crystallisations the material was dissolved in pure water, and to the rather concentrated solution was added a solution of silver nitrate, prepared in the manner already indicated. The white curdy precipitate which separated, after washing with cold water, was dissolved in hot water, the solution filtered and evaporated to crystallisation. The silver acetate separated in brilliant sword-shaped crystals. After pouring off the solution the crystals were quickly rinsed with cold water and placed between filters to remove the adhering moisture. The material was allowed to remain in contact with the filters only for a short time. It was then placed in a platinum dish, and when apparently dry the crystals were broken up into a finely divided condition and dried forty-eight hours in a vacuum desiccator. This work was carried on in a darkened room, and the silver acetate obtained was placed in a weighing tube, and kept in a desiccator in a dark place.

Mode of Procedure.

The method of operation was similar to that described under silver nitrate. After weighing the silver acetate its aqueous or cyanide solution was electrolysed and the weight of the resulting metallic silver determined. The results obtained from the aqueous solution were sometimes vitiated by the separation of silver peroxide at the anode. To prevent this, potassium cyanide was sometimes added. The results, however, from the two solutions were practically the same when no peroxide separated. From the aqueous solution the silver was deposited in a crystalline form. The strength of current and time of action were the same as for silver nitrate.

Ten observations on silver acetate reduced to a vacuum standard on the basis of—

3.241	= density of silver acetate,
10.5	= " metallic silver,
24.4	= " platinum dish,
8.5	= " weights.

and computed for the formula $\text{AgC}_2\text{H}_3\text{O}_2$, assuming the atomic masses of carbon, hydrogen, and oxygen to be 12.01, 1.008, and 16 respectively, are as follows:—

	Weight of $\text{AgC}_2\text{H}_3\text{O}_2$. Grms.	Weight of Ag. Grm.	Atomic mass of silver.
1	0.32470	0.20987	107.904
2	0.40566	0.26223	107.949
3	0.52736	0.34086	107.913
4	0.60300	0.38976	107.921
5	0.67235	0.43455	107.896
6	0.72452	0.46830	107.916
7	0.78232	0.50563	107.898
8	0.79804	0.51590	107.963
9	0.92101	0.59532	107.925
10	1.02495	0.66250	107.923

Mean .. = 107.922

Maximum .. = 107.963

Minimum .. = 107.896

Difference .. = 0.067

Probable error = ± 0.005

Computing from the total quantity of material used and metal obtained, we have 107.918 for the atomic mass of silver.

Experiments on Silver Succinate.

Silver succinate was prepared in a manner similar to that of silver acetate. The commercial C. P. succinic acid was re-crystallised three times; the ammonium salt was then prepared and its aqueous solution precipitated with a solution of pure silver nitrates. The precipitate of silver succinate was thoroughly washed by decantation with pure water and carefully dried. After drying for several hours in an air-bath at 50°, the material was ground in an agate mortar to a finely divided powder, and was then re-dried for twenty-four hours in an air-bath at a temperature of 60°. The white powder obtained in this way was placed in a weighing tube and kept in a desiccator.

The method of analysis was similar to that of silver acetate. A weighed portion of the material was dissolved in a little potassium cyanide in a platinum dish. After diluting with pure water, the solution was electrolysed and the resulting deposit weighed. The strength of current and time of action were the same as for silver nitrate. The results computed for the formula $\text{C}_4\text{H}_4\text{O}_4\text{Ag}_2$ were not constant, and were invariably from one to two units lower than those obtained from silver nitrate and silver acetate. The material was then dried at a temperature of 75°, but the results obtained were not satisfactory.

The two most probable causes for these low results are:—

First, the difficulty of removing the last traces of impurities from a precipitate like that of silver succinate. The experience throughout this work has been, that to remove all the impurities from a finely divided precipitate by washing is almost impossible.

Second, the difficulty met in drying material of this kind. This same difficulty was met in the experiments on silver oxide which, as shown by Lea, retained moisture up to 165°.

THIRD SERIES.

Experiments on Silver Benzoate.

The preceding work on silver acetate and silver succinate shows the necessity of selecting compounds which form well-defined crystals. Perhaps no organic salt of silver fulfils the conditions necessary for accurate analysis better than silver benzoate.

* Contribution from the John Harrison Laboratory of Chemistry No. 13. From the author's thesis presented to the Faculty of the University of Pennsylvania for the degree of Ph.D.—From the *Journal of the American Chemical Society*, xviii., p. 990.

Preparation of Silver Benzoate.

The purest commercial benzoic acid was re-sublimed three times from a porcelain dish into a glass beaker. The product thus obtained was dissolved in pure aqueous ammonia, and the solution evaporated to crystallisation. The ammonium salt was then dissolved in distilled water, and to the solution was added a solution of pure silver nitrate. The white precipitate of silver benzoate which separated was washed with cold water; it was then dissolved in hot water, the solution filtered, and evaporated to crystallisation. The salt separated in fine needles, which clung together in arborescent masses. After removing the liquid from the beaker, the crystals were quickly rinsed with cold water and placed between filters to remove the adhering moisture. When apparently dry they were broken up into small fragments and dried forty-eight hours in a vacuum desiccator. The material was then placed in a glass-stoppered weighing-tube and kept in a dark place.

Mode of Procedure.

The details of the method of operation are the same as those given under silver nitrate. A weighed portion of the material was dissolved in a dilute solution of potassium cyanide in a platinum dish. The solution was then electrolysed and the resulting metal weighed. The strength of current and time of action were the same as for silver nitrate.

Before the results could be reduced to a vacuum standard it was necessary to determine the specific gravity of silver benzoate. This was done by means of a specific gravity bottle, the liquid used being chloroform. The mean of two determinations gave 2.082 for the specific gravity of silver benzoate.

Ten results on this compound, reduced to a vacuum standard on the basis of—

2.082 =	density of silver benzoate,
10.5 =	metallic silver,
21.4 =	platinum dish,
8.5 =	weights,

and computed for the formula $C_7H_5AgO_2$, assuming 12.01, 1.008, and 16 to be the atomic masses of carbon, hydrogen, and oxygen, respectively, are as follows:—

	Weight of $C_7H_5AgO_2$. Gms.	Weight of Ag. Grm.	Atomic mass of silver.
1	0.40858	0.19255	107.947
2	0.46674	0.21999	107.976
3	0.48419	0.22815	107.918
4	0.62432	0.29418	107.918
5	0.66496	0.31340	107.964
6	0.75853	0.35745	107.935
7	0.76918	0.36247	107.936
8	0.81254	0.38286	107.914
9	0.95673	0.45079	107.908
10	1.00840	0.47526	107.962
Mean			= 107.938
Maximum ..			= 107.976
Minimum ..			= 107.908
Difference ..			= 0.068
Probable error			= ± 0.005

Computing from the total quantity of material used and metal obtained, we have 107.936 for the atomic mass of silver.

Summary.

In discussing the work on the atomic mass of silver, two possible sources of error suggest themselves:—

First, the hydrogen which is continually being set free in the process of electrolysis may, in part, be occluded by the metallic silver. As already pointed out, the metallic deposits were washed several times with boiling water, with the hope of removing any occluded gases; but whether this effected a complete removal of all the occluded gases was not determined.

Second, the condensation of moisture on the platinum dish might be urged as a possible source of error. But it must be remembered that the dish was dried in the same manner each time and kept for several hours in a desiccator and that the atmosphere inside the balance was kept dry by means of several beakers of anhydrous calcium chloride, and that the temperature of the balance room throughout the work was almost constant. Under these conditions there is but little chance of error from different amounts of moisture condensed. Moreover, the variation in the different weighings of the same dish was very slight.

The advantages of the method are evident:—

First, the great advantage of the method is its extreme simplicity.

Second, the nature of the compounds used and of metallic silver renders them well adapted to weighing.

Third, the method was such as to eliminate the errors incident to the ordinary gravimetric methods of analysis.

Of the three series, the first is probably entitled to the greatest weight. That the silver nitrate was pure and free from moisture seems beyond question. However, the close agreement of the last two series with the first indicates that the acetate and benzoate of silver were also free from moisture.

Giving equal weight to each of the three series, we have the following as the general mean computed from the separate observation:—

	Atomic mass of silver.
First series	107.924
Second „	107.922
Third „	107.938
General mean =	107.928

Computing the general mean from the total quantities of material used and metal obtained, we have:—

	Atomic mass of silver.
First series	107.926
Second „	107.918
Third „	107.936
General mean =	107.927

Combining this with the first general mean we have 107.9275 as the final result for the atomic mass of silver.

(To be continued).

ALUMINUM ANALYSIS.*

By JAMES OTIS HANDY.

ALTHOUGH the aluminum industry is not a large one in the sense that the iron industry is, it is growing very rapidly. The output of the United States in 1894 was 550,000 pounds, and in 1895 it was about 850,000 pounds. The Pittsburg Reduction Company, with works at New Kensington, near Pittsburg, Pa., and at Niagara Falls, N. Y., is the only American producer of aluminum. The material is made by the electrolysis, in carbon-lined pots, of alumina dissolved in a fused bath of fluorides. The product of each pot is ladled out at intervals, and is graded according to the analyses of the Pittsburg Testing Laboratory, Limited. Some of the aluminum is sold as it is made, and some is alloyed to modify its physical properties. Alloys of aluminum with 3 per cent nickel, or with 3 to 7 per cent copper, or similar amounts of zinc, are very useful, on account of increased strength with only slightly increased specific gravity. The aluminum at present produced with the best ores available contains from—

* From the *Journal of the American Chemical Society*, Sept., 1896.

99 to 99.9 per cent of aluminum.
0.3 to 0.05 per cent of silicon (combined and graphitic).
0.50 to 0.0 per cent of copper.
0.20 to 0.0 per cent of iron.
Carbon is sometimes present in aluminum.

Second grade aluminum contains 96 to 98 per cent aluminum, silicon and iron making up the remainder. Aside from analyses of metallic aluminum, there are required, in the pursuit of the aluminum industry, analyses of alloys of copper, nickel, manganese, chromium, tungsten, zinc, and titanium, with aluminum; aluminum solders, containing tin, zinc, and phosphorus; aluminum hydrate, bauxite, and electrode carbons; hydrofluoric acid and fluorides.

Analysis of Commercial Aluminum. (95 to 99.9 per cent, pure).

In the analysis of aluminum we are offered a choice of solvents.

Solubility of aluminum:—Hydrochloric acid of 33 per cent (*i. e.*, 1 part of hydrochloric acid of 1.2 sp. gr. to 2 parts water) is a rapid solvent.

Sulphuric acid of 25 per cent dissolves aluminum completely on long boiling.

Nitric acid of 1.16 sp. gr. dissolves aluminum on prolonged boiling.

Acid mixture:—A mixture of the three acids, which we term "Acid Mixture" is made of—

100 c.c. nitric acid of 1.42 sp. gr.
300 c.c. hydrochloric acid of 1.20 sp. gr.
600 c.c. sulphuric acid of 25 per cent.

It is a very useful solvent for aluminum, because it supplies oxidising conditions during solution and so prevents loss of combined silicon as hydride. The sulphuric acid content of the acid mixture furnishes a means of rapidly dehydrating the silica.

Sodium hydroxide solution of 33 per cent is a useful solvent when it is desired to separate the metallic impurities from the bulk of the aluminum at once. Weaker solutions do not work as quickly or completely. Solution succeeds best in an Erlenmeyer flask.

Fifteen c.c. of the sodium hydroxide solution suffice for 1 gm. of aluminum.

Commercial soda lye may be used if dissolved and filtered through asbestos.

Other Reagents and Standard Solutions used in Aluminum Analysis.

Sodium carbonate, chemically pure.

Soda ash: "Solvay" soda ash, a saturated solution in water, filtered.

Powdered zinc: practically free from iron and copper.

Fifteen per cent nitric wash: (15 parts 1.42 nitric acid to 85 parts water).

Standard potassium permanganate: 5.76 grms. in 2 litres. One c.c. equals 0.005 gm. iron.

Standard potassium cyanide: 45 grms. in 2 litres. One c.c. is made to equal 0.005 gm. copper.

Special Apparatus.

Two narrow glass tubes, graduated roughly, one to hold 1 gm. of powdered zinc, and the other 1 gm. of chemically pure sodium carbonate.

The evaporating dishes which are used are, preferably, about 4½ inches in diameter, and are covered with 5-inch glasses.

The Erlenmeyer flasks are of about 12-ounce capacity, and furnished with porcelain crucible covers.

The Method.

Determination of Silicon, Iron, and Copper in Commercial Aluminum.—One gm. of aluminum drillings is dissolved in a 4½-inch evaporating dish in 30 c.c. of "acid

mixture." If the drillings are thin it is best to add only 15 c.c. at first. Placing cold water on the cover-glass sometimes prevents loss from too energetic foaming. Solution having been completed by warming slightly, evaporate quickly to strong fumes of sulphuric acid, and continue heating for five minutes. Experience will show on what parts of the hot plate these solutions can be evaporated without spattering at the time when aluminum sulphate begins to crystallise out. Remove the dishes from the plate by means of an iron fork, and in a few moments add to the contents of each 75 to 100 c.c. of water and 10 c.c. of 25 per cent sulphuric acid, break up the residue in each dish with a short heavy glass rod, and place the dishes back on the hot plate. Boil until all aluminum sulphate dissolves. Add to each dish 1 gm. of metallic zinc powder, measured. Be careful to pour the zinc into the middle of the liquid. If it touches the sides of the dish it sometimes becomes firmly fixed there. Keep the dish contents at 60° to 70° C. until the zinc has dissolved, leaving the iron reduced and the copper precipitated. Filter and wash well with hot water. Cool, titrate the filtrates with standard potassium permanganate. One c.c. equals 0.50 per cent iron when 1 gm. of aluminum has been used. Placing new receivers under the funnels, treat each residue twice with hot 15 per cent nitric acid wash. Wash out with water, and in the solutions thus obtained titrate the copper with standard potassium cyanide, after adding saturated soda ash solution until the precipitated copper carbonate re-dissolves. The end point of the titration is very satisfactory. The cyanide solution should be standardised with copper of known purity in about the amount usually found, *viz.*, 0.005 to 0.010 gm. The residue of silicon and silica are burned off in numbered crucibles, and each fused with 1 gm. of chemically pure sodium carbonate (measured). The crucible containing the fused mass is placed in 15 c.c. of water in the porcelain dish originally used, and 25 c.c. of 25 per cent sulphuric acid are added. Solution takes place quickly without separation of silica, and after rinsing out and removing the crucible, the solution is evaporated to five minutes fuming on the hot plate. After cooling add 75 to 100 c.c. of water, and boil to disintegrate the silica. Filter and wash well with water. Burn off and weigh silica and crucible, treat with hydrofluoric acid and a drop of sulphuric acid if impurity is suspected. Evaporate, ignite, and weigh again. Loss equals silica; calculate to silicon.

Determination of Crystalline (Graphitic) Silicon in Aluminum.—Dissolve 1 gm. of aluminum in 30 c.c. of 33 per cent hydrochloric acid (two parts of water to one of hydrochloric acid) in a platinum dish; add about 2 c.c. of hydrofluoric acid, stir, and filter at once through a No. 0 9 c.m. filter, contained in a funnel which has been thinly coated with paraffin. Wash with water and burn off in a platinum crucible. Fuse with 1 gm. of sodium carbonate, cool in 15 c.c. of water in a four-and-a-half-inch evaporating dish. Add 20 c.c. of 25 per cent sulphuric acid. Rinse out the crucible. Evaporate to fumes, cool, add 75 c.c. of water, boil up, and filter off the silica. Wash, ignite, and weigh. Calculate to silicon.

The determinations of silicon, copper, and iron are the every-day methods of grading aluminum. It is recognised that sodium and carbon occasionally exist in aluminum, and they are determined by methods described. In certain samples it is desirable to know the approximate percentage of graphitic and combined silicon. These determinations are also described. We determine nitrogen, if present, by a special method.

(To be continued).

The Chemical Society.—At the last meeting of the Chemical Society it was announced that Mr. J. J. Tustin had made a donation of one thousand guineas to the Research Fund of the Society.

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, January 22nd, 1897.

Prof. AYRTON, Vice-President, in the Chair.

MR. CROFT gave an exhibition of some simple apparatus.

The exhibition included an ingenious form of clip to fit on an upright retort stand; a nicol used for projecting the rings and brushes in crystals, with which it is sufficient to use the ordinary condenser of the lantern, the source of light having been moved further away from the lens than is usual; some photographs showing caustics, conical refraction, and diffraction; a stand for magnets, &c., when demonstrating the attraction and repulsion of poles; a stand for the suspension of objects for experiments on diamagnetism; a holder for X ray tubes, consisting of a spiral of wire fitting round the exhaustion tube of the bulb; an X ray photograph taken by means of a Wimshurst machine; a model of Michelson's interference experiment; an arrangement to show subjective colours, in which a double lantern is arranged to give two partly overlapping discs. A sheet of green glass is placed before one lantern, and the light of the other decreased till the illumination of the two discs is the same. The overlap then appears white, while the remainder of the uncoloured disc appears red.

Prof. SILVANUS THOMPSON said he was surprised that "patent plate" was sufficiently good for Michelson's experiment. Had the author tried illuminating the discs in his subjective effect experiment for a very short interval, so that the eye would not have time to wander from one disc to the other?

MR. GRIFFITH said that if you looked through a tube at one disc at a time, one appeared green and the other white.

The CHAIRMAN said the point seemed to be, could you fatigue the eye simultaneously, or must it be successive?

Prof. SILVANUS THOMPSON said two common one-inch microscope objectives were very suitable for projecting rings and brushes.

MR. E. C. BALLY read a paper on the "*Passage of Electricity through Gases*."

In this paper, which is of a purely controversial nature, the author brings forward as arguments that electrical conduction in gases is not of an electrolytic nature the following:—(1) That the sign of the supposed gaseous ion is variable. (2) The initial resistance of a gas. (3) The invalidity of Ohm's law. (4) The permanence of the supposed gaseous electrolyte. (5) That every mixture of gases must equally be an electrolyte. (6) That the potential gradient in a vacuum-tube when the current is passing has been shown to be very uneven. It is very steep in the cathode glow, and is by no means a regular decline between the electrodes.

Prof. ARMSTRONG said it was difficult to know from what point of view the author had treated the question. The first part of the paper consisted almost entirely of a criticism of Prof. J. J. Thomson's theory and experiments. Prof. Thomson, however, is not the only observer who has dealt with this subject. The author's arguments seemed vitiated by the fact that he has looked upon the subject from one very narrow standpoint only, viz., the ionic hypothesis, and Lord Kelvin, for instance, does not believe in the truth of the ionic hypothesis even in the case of liquids. Prof. Thomson has shown that the phenomena depend on the dryness of the gas, so that the conduction cannot depend on the gaseous molecule alone. In the case of conduction induced by a neighbouring discharge, this might be due to the expulsion of condensed vapour from the walls of the vessel. It would appear that in the dry state gases are not electrolytes.

MR. ENRIGHT said he thought it was not correct to say no work was done in electrolysis.

Prof. SILVANUS THOMPSON said that the pursuit of the analogy between the conductivity in gases and liquids was apt to lead one too far. Thus, if you compare the conduction in a mixture of H and Cl with electrolysis, your analogy will be a false one unless you import into the term electrolysis the idea of chemical separation as taking place in the solution. If a current separated a mixture of powdered zinc and sulphur, it could not be called a case of electrolysis.

Prof. ARMSTRONG said an experiment of Prof. Dewar's was very instructive. He had shown that if you cool the surface of a Crookes tube the discharge stops. It was quite inconceivable that at these low pressures the gas became liquefied, so that this experiment seemed to show that conductivity depends on the presence of a vaporous electrolyte.

MR. ENRIGHT asked if Prof. Armstrong knew how the presence of an electrolyte assisted conduction.

In a communication Prof. J. J. THOMSON said that in the decomposition of steam by a spark, the fact that in the tube as a whole the amount of steam decomposed is greater than the amount of gases liberated in a voltmeter in series was no objection to the conductivity being electrolytic. The only condition imposed by the laws of electrolysis was that the excess of H or O at one terminal, and of O or H at the other, should correspond to the amount of electricity passing through the tube. Thus, suppose in a water voltmeter a number of metal partitions are fixed so that the current has to pass across these plates. Then at each plate H will be given off on one side and O on the other, and by making the partitions sufficiently numerous the total quantity of gases given off for the passage of a given current may be made as large as we please. The excess at the terminals would not be affected at all by these partitions. In the experiments made by Mr. Rutherford and himself (Prof. Thomson), they did not observe any polarisation when the conductivity was produced by Röntgen rays. With reference to Mr. Baly's objections to the electrolytic theory, (1) There is no reason to think that under conditions other than in solution the atom of hydrogen may not have a negative charge. (2) The electrolytic theory leads us to expect that it would require a finite electromotive force to send a discharge through a gas. Before such a discharge can take place, the molecules must be split up, and this requires an electric field of finite strength. (3) In the case of a gas the electric field has to ionise the molecules, so that an increase in the strength of the field will not only (as in the case of a liquid electrolyte) increase the speed of the ions, but it will also increase their number, and thus the current will increase faster than the electromotive force. (4) The ion once used can again combine, and since the ionisation is done by the electric field it can be again split up and used again. If, however, the ionisation has been done by external sources, as, for example, by Röntgen rays, then we find that the conductivity decreases as the current passes. (5) There seems to be no reason on the electrolytic theory why in a mixture of HCl and Cl some of the current should not go through the chlorine. (6) A variable potential gradient would be produced if the ions moved with different velocities. Mr. Baly's process in the positive column appears to be the same as on the electrolytic theory, minus the atomic charges.

In a communication Prof. SCHUSTER said: Mr. Baly criticises what he calls the electrolytic theory, but directs his arguments against a form of the theory which is, as far as the writer knows, upheld by no one. Mr. Baly appears not to have read the original papers in which the fundamental points of the theory upheld by J. J. Thomson and the writer (Prof. Schuster) are explained. If he had done so he could not have given as an objection to the theory that the conductivity of a gas increases with the E.M.F. The essential difference between a liquid

and a gas is that in the liquid the number of ions is fixed by the chemical constitution of the liquid, while in a gas dissociation has first of all to be produced by the current itself, and hence the number of ions depends on the current. In the paper referred to by Mr. Baly, in which the fact that when a spark is passed through a gas the gas ceases to insulate for some distance round the spark is described, the explanation that this was due to a difficulty of passage of the electricity from the electrode into the gas was especially disclaimed, the explanation given being substantially the same as that now given by Mr. Baly. Mr. Baly asks what becomes of the ions that are set free? The answer, of course, is that they re-combine. The view that stratifications are due to compound molecules, and do not probably occur in pure gases, is not new. With reference to the author's statement about "measurements made by Wheatstone and J. J. Thomson prove that the electricity travels along the positive column from the anode to the cathode, and that its velocity is about half that of light," Prof. Thomson's results show that the breakdown of the insulating power of air takes place in the manner described, but this does not show anything as to what happens when the discharge has reached the steady state. Mr. Baly is quite wrong in the excess charges he assigns to different parts of the vacuum tube. Experiments on the excess charges can count for nothing unless they are done with continuous currents. Mr. Baly is further wrong in stating that the fall of potential is rapid in the glow; on the contrary, it is very small in the glow, being very rapid in the dark space between the glow and the cathode. Mr. Baly adopts Prof. Thomson's view as to the formation of molecular chains, but in a form very difficult to accept. The whole foundation of Mr. Baly's theory is upset by his wrong assumptions as to the excess charges in different parts of the tube.

The AUTHOR, in his reply, said, that on some points he had been misunderstood. He thought that the increase in conductivity could not be due to vapour driven off from the sides, for ultra-violet light also produced such an increase. If Röntgen rays produce ionisation, then there ought to be a reduction in the density of the gas.

NOTICES OF BOOKS.

Röntgen Rays, and Phenomena of the Anode and Cathode.
By EDWARD P. THOMPSON, M.E., E.E. With concluding Chapter by Prof. WILLIAM A. ANTHONY. New York: D. Van Nostrand Company.

THIS book is really a collection of abstracts from papers dealing with electrical discharges generally, and with the production of X rays in particular; it is well printed, and the illustrations are good, but there seems to be an idea in the mind of the printer that these latter need to be uniformly distributed through the book, with the result that within the first 70 pages or so we have no less than seven illustrations all referring to matter beyond pages 100. The first 60 pages are occupied with details of experiments on anodic and cathodic phenomena made long previous to Röntgen's discovery, the reason for this being, as the author points out in his preface, "that the student and general reader, whose object is to become acquainted with the properties of cathodic and X rays, might better understand them." This is very commendable, only we are rather afraid that the general reader will be in danger of confusion, while for the student the abstracts are far too meagre to be of much value beyond that of an index to the publications.

The book contains far too much of the sensational newspaper character that seems quite out of place; for instance, a whole-page illustration of "Edison's beneficent X-ray exhibit" at the Electrical Exposition in 1896 is given on page 37, and is referred to on page 71 as

an instance when "thousands of people, through the beneficence of Dr. Edison, were permitted to see the shadows of their bones surrounded by living flesh!"

How this will enable either students or general readers to become better acquainted with X rays is difficult to comprehend.

Another personal illustration, given on page 122, seems equally superfluous; in this a Sprengel pump is shown of such an obsolete form, that if the physicist in question really used such an apparatus the wonder is that he ever produced any successful results at all.

The abstracts of the most important papers are good. The review of Spottiswoode and Moulton's paper on Sensitive Discharges, Mr. Crookes's on Radiant Matter, and Lenard's Experiments, show that the author has thoroughly investigated the subject; but again we must point out that they seem too technical for the general reader, and the student had far better refer to original papers for an intelligent knowledge of the experiments and the conclusions drawn from them. This cannot be got from an abstract, no matter how carefully it may have been made.

In several places there are statements that cannot fail to mislead an uninformed reader. For instance, on page 47 Perrin's experiment to find out if the cathodic rays carry negative charges is given with illustrations, but it is not stated that this was fully demonstrated five years previously by Mr. Crookes in his Address before the Institute of Electrical Engineers.

On page 96 a standard X-ray tube is shown with two concave cathodes, one at either end, and this is stated to have been "first proposed by Prof. Elihu Thompson." As this form of tube immediately suggested itself to many workers on the subject, it was proposed by numbers of people in the early days of X-ray work, and it is difficult to see the value of a claim to priority, especially in view of the fact that it does not appear to have fulfilled the hopes of those who proposed it. With regard to the so-called "focus tube," which is really an unimportant modification of Mr. Crookes's incandescent platinum tube, it is said that King's College published a description of it, and that Mr. Shallenberger was the first, as far as the author knew, to originate it. It is perfectly well known in England, at least, that the use of this tube was fully described by Mr. Jackson, of King's College, to whose energy much of the subsequent success of practical skiagraphy is due, and it seems a pity that his name is not given.

Great prominence is given to the calcium-tungstate screen for X-ray work. This material, although it possesses the advantage of cheapness, is much inferior to barium or potassium platino-cyanide.

On the whole, we cannot but feel disappointed that the book, which, as the author says, is got up regardless of expense, should be of no more use than a compilation of abstracts.

CORRESPONDENCE.

REPORT OF COMMITTEE ON THE PROXIMATE CONSTITUENTS OF COAL.

To the Editor of the Chemical News.

SIR,—In the CHEMICAL NEWS (vol. lxxiv., p. 292) there appeared a letter from Messrs. Cross and Bevan relating to the above report. To this I should have replied before had not my vacation intervened, during which I was unable to refer to the papers cited in the letter.

As Secretary of the Committee, and mainly responsible for the drawing up of the Report, I would wish, in the first place, to take the onus of the shortcomings of the Report on my own shoulders, and, secondly, to disavow entirely any intention to disparage or belittle in any way

the work of Messrs. Cross and Bevan on this subject. That the scope of their investigations had not been properly appreciated by me arose entirely from the fact that I was not—as I ought certainly to have been—aware of the extent of their investigations. The perusal of their papers published in 1881, and in the *Philosophical Magazine* of 1882, has led me to realise something of what they have done toward furthering our knowledge of the relations of coal. I wish, therefore, to acknowledge the justice of their claim to priority for the method of attacking this problem by the action of chlorinating agents.—I am, &c.,

P. PHILLIPS BEDSON.

The Durham College of Science,
Newcastle-upon-Tyne, January 20, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 2, January 11, 1897.

Obituary.—M. Loewy gave an account of the career and researches of Prof. B. A. Gould, the illustrious astronomer, of Cambridge (U.S.A.), who died on November 26th last. The deceased was one of the first who successfully applied photography to the determination of the positions of the stars.

The Academy proceeded to nominate a Commission charged with the appointment of a young French *savant*, to whom will be granted the "encouragement" founded by the Royal Society of London in memory of the eminent physicist Joule.

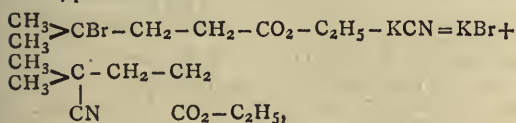
M. Berthelot presented to the Academy a volume entitled "*Serie intorne alla Teorie Moleculare ad Atomica ad alla Notazone Chimica*," by S. Cannizzaro. This volume has been printed by occasion of the 70th anniversary of the birth of the eminent chemist, at the expense of an international subscription (July 13, 1896).

Density of Ozone.—Marius Otto.—The density is $\frac{1}{2}$ times that of oxygen, *i.e.*, 1.6584.

Decomposition of Metallic Sulphates by Hydrochloric Acid.—Albert Colson.—If the action of hydrochloric acid upon sulphates is assimilable to heterogeneous dissociations two conclusions are necessary:—1. Sulphuric acid at about 15° will not attack lead chloride placed in an atmosphere of hydrochloric acid gas. The attack takes place only if the pressure is sufficiently reduced. Not merely is the principle of mechanical equivalence applicable to these phenomena of displacement, but Carnot's principle intervenes in a decided manner.

Polymerisation of some Cyanic Compounds, being a Rectification of some of the Author's former Paper on Cy_3Cl_3 .—Paul Lamoult.—A thermo-chemical paper.

Action of Potassium Cyanide upon the Olides 1-4.—Edmond Blaise.—The author is seeking to effect the synthesis of dimethyl 2-2 pentanedioic acid by the action of potassium cyanide upon bromo -2-, ethyl -α-, pentanoate—



and saponification of the nitrile ether obtained.

Phosphoric Ethers of Allylic Alcohol.—J. Cavalier.—The author is studying the monoallyl-phosphoric, diallylic and triallyl-phosphoric acids.

A Difference between the "Top" and "Bottom" Yeasts.—P. Petit.—Top yeast consumes more than double the amidic nitrogen of bottom yeast, and, on the contrary, much ammoniacal nitrogen.

MEETINGS FOR THE WEEK.

MONDAY, Feb. 1st.—Society of Arts, 8. (Cantor Lectures). "Material and Design in Pottery," by Wm. Burton, F.C.S.

TUESDAY, 2nd.—Royal Institution, 3. "Animal Electricity," by Prof. A. D. Waller, F.R.S.

Society of Arts, 8. "The Progress of the British Colonial Empire during the past Sixty Years of Her Majesty's Reign," by the Right Hon. Sir Charles W. Dilke, Bart., M.P.

WEDNESDAY, 3rd.—Society of Arts, 8. "The Recommendations of the Recess Committee for the Development of Ireland's Industrial Resources," by The Right Hon. Horace Plunkett, M.P.

Society of Public Analysts, 8. "The Composition of Meat Extracts and similar Products," by Otto Hohner. "The Distillation of Formaldehyd from Aqueous Solution," by Norman Leonard, B.Sc., Harry M. Smith, and H. Droop Richmond. "Some Analyses of Water from an Oyster Fishery," "Remarks on Formaldehyd" (postponed from last meeting), by Charles E. Cassal.

THURSDAY, 4th.—Royal Institution, 3. "Some Secrets of Crystals," by Prof. H. A. Miers, F.R.S.

Society of Arts, 8. "The Mechanical Production of Cold," by Prof. James A. Ewing, M.A., F.R.S.

Chemical, 8. "The Oxidation of Nitrogen," by Lord Rayleigh. "Researches in the Stilbene Series, I.," by J. J. Sudborough, Ph.D. "Diortho-substituted Benzoic Acids. III. Hydrolysis of Substituted Benzamines," by J. J. Sudborough, Percy G. Jackson, and L. L. Lloyd. "Apparatus for Steam Distillation," by F. E. Matthews, Ph.D. "Oxidation of Sulphurous Acid by Potassium Permanganate," by T. S. Dymond and F. Hughes.

FRIDAY, 5th.—Royal Institution, 9. "The Picturesque in History," by The Right Rev. The Lord Bishop of London.

SATURDAY, 6th.—Royal Institution, 9. "Neglected Italian and French Composers," by Carl Armbruster.

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(SYNOPTICAL CIRCULAR ON APPLICATION).

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1941.

ON SOME CHROMATIC REACTIONS PRODUCED BY ORGANIC ACIDS, PRINCIPALLY TARTARIC, CITRIC, AND MALIC ACIDS.

By E. PINERNA,
Professor of Chemistry in the University of Valladolid.

THE reagent employed by me contains of a fresh solution of naphthol-β in concentrated sulphuric acid (β-naphthol-sulphuric acid). It is prepared with 0.02 grm. of naphthol-β and 1 c.c. of sulphuric acid, 1.83.

Five centigrms. of each of the organic acids, or the residue left on the evaporation of their solutions, gradually heated in a little porcelain capsule with a spirit flame, after having added from 10 to 15 drops of the reagent, produce the following tints:—

Tartaric acid, pure, gives at first a blue colour, and on continuing gradually to heat, it produces a very decided green colouration. On adding water to the liquid resulting from the reaction, after it has cooled (15 to 20 times its volume), the colour changes to a persistent reddish-yellow.

Citric acid, pure, gives an intense blue colour which does not change to a bright green even when it is heated gently for a long time. On pouring fifteen or twenty times its bulk of water on the liquid after cooling, the solution remains colourless or takes a light yellow colour. A small quantity of tartaric acid added to the citric acid is sufficient to produce the above-mentioned indigo-green colour.

With pure citric acid we never obtain the strong green colouration so characteristic of tartaric acid. A dull blue-green corresponds to the presence of 10 or 12 per cent of tartaric acid.

Malic acid, pure, first gives a greenish yellow colour, and on continuing the gradual action of heat a bright yellow colour. When the amount of acid is very small the colouration is well seen by shaking the capsule so that the liquid resulting from the reaction moistens the interior side, and the portion which remains on the sides is of a very perceptible yellow colour. The addition of water changes the colour to a bright orange.

All the reactions of these organic acids are characteristic, and can be produced with great facility.

It is only necessary to pay great attention to the employment of heat, and to notice the moment when any colouration commences, and then to remove the capsule from the fire, waiting till the change has terminated before re-commencing to heat (if necessary) until there appear the colours corresponding to each of the acids indicated above.

The reactions produced by other organic acids are different in colour and tint, but are not so characteristic or brilliant as those already pointed out. One must proceed to separation by solvents of the colouring matters produced. Other coloured reactions are produced with nitrites, nitrates, and chlorates. On adding ten drops of the above described reagent to five centigrms. of sodium nitrite with three or four drops of water, a very strong red colouration is produced, and the addition of water does not change it.

On pouring ten drops of a solution of resorcin in sulphuric acid (0.1 grm. of resorcin in 1 c.c. of sulphuric acid at 66°) on five centigrms. of sodium or potassium nitrate, there is produced at first a red-brown colouration, and then a very intense violet, changing to orange on the addition of water.

With potassium chlorate (two centigrms. of chlorate suffices) a very intense green colour is produced, changing to brown on the addition of water.

THE DETERMINATION OF ATOMIC MASSES OF SILVER, MERCURY, AND CADMIUM, BY THE ELECTROLYTIC METHOD.*

By WILLETT LEPLEY HARDIN.

(Continued from p. 55).

PART II.

Determination of the Atomic Mass of Mercury.

From all the earlier determinations Clarke gives 200 as the most probable value for the atomic mass of mercury, assuming oxygen equal to 16.

Experiments on Mercuric Oxide.

A large number of experiments were made with a view of determining the ratio of mercury to oxygen in mercuric oxide. The method proved to be unsatisfactory, although apparently very good results were obtained in some preliminary experiments. The cause of this close agreement of results will be explained in the details of the work.

Preparation of Pure Mercuric Oxide.

The purest commercial mercuric chloride was carefully sublimed from a porcelain dish into a glass funnel. The sublimed portion was dissolved in water, the solution filtered, and evaporated to crystallisation. The crystals were then thoroughly dried and carefully re-sublimed. The product obtained in this way consisted of white crystalline leaflets which dissolved completely in water. Pure sodium hydroxide was then prepared by throwing pieces of metallic sodium on pure water contained in a platinum dish. To the pure sodium hydroxide was added a solution of mercuric chloride, the former always being in excess. The yellow mercuric oxide which separated was washed for several days by decantation with hot water. The material was then dried twenty-four hours in an air bath at 105°.

Mode of Procedure.

In a series of preliminary experiments made in the spring of 1895, a weighed portion of mercuric oxide prepared in the above manner was dissolved in a dilute solution of potassium cyanide in a platinum dish. The solution was then electrolysed and the weight of the resulting metallic mercury determined. Inasmuch as the results obtained in these preliminary experiments were not reduced to a vacuum standard, it was thought advisable to weigh the empty platinum dish after removing the metallic deposit in order that the two weighings might be made under approximately the same conditions. The results for the most part agreed very closely and differed very little from the results obtained by other methods. Six observations computed for the formula HgO, assuming the atomic mass of oxygen to be 16, are as follows:—

Atomic Masses of Silver, Mercury, and Cadmium.

	Weight of HgO. Grm.	Weight of Hg. Grm.	Atomic mass of mercury.
1	0.26223	0.24281	200.05
2	0.23830	0.22065	200.02
3	0.23200	0.21482	200.06
4	0.14148	0.13100	200.00
5	0.29799	0.27592	200.03
6	0.19631	0.18177	200.02

Mean=200.03.

* Contribution from the John Harrison Laboratory of Chemistry No. 13. From the author's thesis presented to the Faculty of the University of Pennsylvania for the degree of Ph.D.—From the *Journal of the American Chemical Society*, xviii., p. 990.

These results were selected from a larger series. After making the above observations it was noticed that the platinum dish had gradually decreased in weight throughout the work. This decrease in weight indicated that the mercury deposit had formed an amalgam with the platinum dish, which was soluble in hot nitric acid. To ascertain whether such was the case or not the platinum dish, after weighing, was filled with a solution of the double cyanide of mercury and potassium and the solution electrolysed. On dissolving the mercury deposit in cold nitric acid a dark coloured film remained on the sides of the dish. The dish was then carefully washed, dried, and re-weighed, and found to be heavier than at the beginning of the operation, showing that the mercury had not been completely removed. The dark film was then dissolved in hot nitric acid and the dish again weighed. This last weight being less than that at the beginning showed that some of the platinum had been dissolved from the dish. The nitric acid solution of the dark film was evaporated to dryness and ignited to remove the mercury. The residue was dissolved in aqua regia, the solution evaporated to dryness, and enough water added to dissolve the small residue. A little concentrated ammonium chloride was then added to the solution, and the double chloride of ammonium and platinum separated as a yellow crystalline powder. This proved conclusively that the mercury deposit had united with the platinum dish to form an amalgam which was soluble in hot nitric acid. Hence the results given for mercuric oxide are of no value in determining the atomic mass of mercury.

A series of careful experiments was then made on the oxide dried at different temperatures. To avoid any error from the amalgam which formed with each deposit, the platinum dish was weighed at the beginning of each observation, the temperature and barometric pressure being noted at the same time. The results obtained from the oxide dried at a temperature of 105° gave from 180 to 185 for the atomic mass of mercury. The material was then dried at a temperature of 125° , but the increase in the amount of mercury obtained was very slight. Finally, with material dried at 150° , the results obtained for the atomic mass of mercury were all below 195° .

The most probable causes for these low results are:—

First, the difficulty of removing the last traces of alkalis from the mercuric oxide.

Second, the difficulty met in the complete removal of the moisture from an amorphous precipitate. This difficulty as well as the first was referred to in the experiments on silver oxide.

Third, mercuric oxide does not form a clear solution with potassium cyanide. There seems to be a slight reduction of the oxide to the metallic state. It is difficult to determine whether this reduced portion unites completely with the metallic deposit or is partially removed in the process of washing. The latter is probably true, and it may be that a different method of analysis would give more accurate results for this compound.

FIRST SERIES.

Experiments on Mercuric Chloride.

The material used in this series of experiments was prepared from the commercial C. P. mercuric chloride. The product was first dissolved in water, the solution filtered and evaporated to crystallisation. The crystals were dried and carefully sublimed from a porcelain dish into a glass funnel. The sublimed portion was dissolved in water, the solution filtered and evaporated to crystallisation. These crystals were dried as before and carefully re-sublimed. The material was then placed in a weighing tube and kept in a desiccator.

Mode of Procedure.

The method of operation was similar to that already described under the different compounds of silver. A weighed portion of the mercuric chloride was dissolved in a little potassium cyanide and the solution electrolysed.

The deposit was washed and dried and handled in every way like the deposits of silver. The strength of the current and time of action were as follows:—

Time of action.	Strength of current.
4 hours	N.D. ₁₀₀ =0.02 amperes.
6 "	N.D. ₁₀₀ =0.05 "
6 "	N.D. ₁₀₀ =0.10 "
6 "	N.D. ₁₀₀ =0.30 "

A current of gradually increasing strength deposits the mercury in extremely small globules, which can be washed and handled more easily than the larger globules obtained by using a strong current at first. In cases where more than one-half grm. of metal was deposited the strong current was allowed to act two hours longer.

Ten results on mercuric chloride reduced to a vacuum standard on the basis of

5.41 = density of mercuric chloride,
13.59 = " " metallic mercury,
21.4 = " " platinum dish,
8.5 = " " weights,

and computed from the formula HgCl_2 , assuming 35.45 to be the atomic mass of chlorine, are as follows:—

	Weight of HgCl_2 . Grms.	Weight of Hg. Grm.	Atomic mass of mercury.
1	0.45932	0.33912	200.030
2	0.54735	0.40415	200.099
3	0.56002	0.41348	200.053
4	0.63586	0.46941	199.947
5	0.64365	0.47521	200.026
6	0.73281	0.54101	199.988
7	0.86467	0.63840	200.838
8	1.06776	0.78825	199.946
9	1.07945	0.79685	199.917
10	1.51402	1.11780	200.028
Mean			200.006
Maximum			200.099
Minimum			199.917
Difference			± 0.182
Probable error =			± 0.011

Computing from the total quantity of material used and metal obtained we have 199.996 for the atomic mass of mercury.

SECOND SERIES.

Experiments on Mercuric Bromide.

The bromine used in these experiments was prepared by distilling the commercial C. P. bromine twice over manganese dioxide. Any trace of chlorine which might be present would be removed by this method.

Preparation of Mercuric Bromide.

Fifty grms. of metallic mercury were placed in a beaker and covered with water. Pure bromine was then added until the mercury was completely saturated. The contents of the beaker were then digested with hot water until the mercuric bromide dissolved; the solution was filtered and evaporated to crystallisation. The white crystals of mercuric bromide which separated were thoroughly dried and carefully sublimed from a porcelain dish into a glass funnel. Only the middle portion of the sublimate was used in the experiments. The product obtained in this way consisted of brilliant crystalline leaflets which dissolved completely in water. The material was kept in a weighing tube in a desiccator.

Mode of Procedure.

The method of analysis was exactly like that described under mercuric chloride. A weighed portion of the mercuric bromide was dissolved in dilute potassium cyanide in a platinum dish. The solution was then electrolysed

and the resulting metal weighed. The strength of current and time of action were the same as for mercuric chloride.

Ten results on mercuric bromide reduced to a vacuum standard on the basis of

5.92 =	density of mercuric bromide,
13.59 =	" metallic mercury,
21.4 =	" platinum dish,
8.5 =	" weights,

and computed for the formula $HgBr_2$, assuming 79.95 to be the atomic mass of bromine, are as follows:—

	Weight of $HgBr_2$. Grms.	Weight of Hg. Grm.	Atomic mass of mercury.
1	0.70002	0.38892	199.898
2	0.56430	0.31350	199.876
3	0.57142	0.31750	199.938
4	0.77285	0.42932	199.832
5	0.80930	0.44955	199.814
6	0.85342	0.47416	199.911
7	1.11076	0.61708	199.869
8	1.17270	0.65145	199.840
9	1.26186	0.70107	199.899
10	1.40142	0.77870	199.952

Mean ..	=	199.883
Maximum ..	=	199.952
Minimum ..	=	199.814

Difference ..	=	0.138
Probable error	=	± 0.010

Computing from the total quantity of material used and metal obtained, the atomic mass of mercury is 199.885.

(To be continued).

METAL SEPARATIONS BY MEANS OF HYDROCHLORIC ACID GAS.*

By J. BIRD MOYER.

(Continued from p. 54).

VI.—Behaviour of Cupric Oxide.

PURE copper nitrate was made by re-crystallisation. It was then ignited in a porcelain crucible at a dull red heat, until it became constant in weight. The pure black oxide was then subjected to the action of hydrochloric acid gas. In Experiment I., the boat containing the oxide was heated at the outset to 175° . It was taken out after two hours, placed over sulphuric acid for half-an-hour, and weighed. The weight showed that the copper oxide had hardly been acted upon. It had only been superficially changed to chloride. It was then moistened with two or three drops of hydrochloric acid, dried in a rapid current of the gas, and heated two hours longer. This resulted in the complete transformation into chloride. The anhydrous chloride thus obtained, liver brown in colour, was placed in a desiccator from which the air was exhausted. This was done to remove all the gas that might be retained, and prevented a too rapid absorption of moisture.

Copper chloride absorbs moisture, but not so rapidly as to prevent weighing in this form:—

	Copper oxide taken. Grm.	Copper chloride obtained. Grm.	Copper chloride required. Grm.	Difference. Grm.
Experiment I.	0.1011	0.1708	0.1713	-0.0005
" II.	0.1025	0.1726	0.1736	-0.0010
" III.	0.1034	0.1756	0.1752	+0.0004

In Experiment II. the change was completed in the cold by prolonged action through four hours. It was then heated about ten minutes at the end to drive out the moisture that had formed. In all the experiments cited, the copper chloride, after weighing, was found to dissolve completely in cold water.

VII.—The Separation of Antimony from Copper.

The same material was used as in the preceding experiments.

The weighed oxides were thoroughly mixed. The antimony was completely volatilised, leaving copper chloride which was weighed as such. The volatile antimony chloride was caught in the bulb receiver at the end of the tube. The bulb and tube were washed out with acidulated water into a beaker, and the antimony thrown down with hydrogen sulphide. The antimony sulphide was filtered, thoroughly washed, and while moist dissolved in strong hydrochloric acid. The hydrogen sulphide evolved was conducted into bromine water and oxidised to sulphuric acid, which was estimated as usual and the antimony calculated.

The length of time required was eight hours. On several occasions the experiment was interrupted at the end of four hours, but invariably the separation was incomplete, and on dissolving out the copper chloride formed, black copper oxide and white antimony oxide were plainly evident. In some cases the mixture of oxides was moistened with a couple of drops of hydrochloric acid, and then evaporated down in a stream of acid gas by heating the tube over a water-bath. This treatment seemed to facilitate matters, but it is not altogether advisable, because the copper chloride has a tendency to creep over the sides of the boat. It is quicker in the end to separate them in the dry condition, allowing plenty of time for the reaction. The copper chloride obtained was perfectly soluble in cold water and contained no antimony. It could readily be changed to oxide and weighed if thought necessary.

	Antimony trioxide taken. Grm.	Copper oxide taken. Grm.	Copper chloride obtained. Grm.	Copper chloride required. Grm.	Difference. Grm.
Experiment I.	0.1068	0.1040	0.1750	0.1745	+0.0005
" II.	0.1062	0.1053	0.1774	0.1784	-0.0010
" III.	0.1022	0.1020	0.1726	0.1728	-0.0002
" IV.	0.1198	0.1020	0.1722	0.1723	-0.0006

	Antimony trioxide taken. Grm.	Antimony trioxide found. Grm.	Difference. Grm.
Experiment I.	0.1068	0.1059	+0.0009

VIII.—The Separation of Bismuth from Copper.

The pure oxides were mixed and treated as directed under bismuth and lead.

	Copper oxide taken. Grm.	Bismuth trichloride taken. Grm.	Copper chloride obtained. Grm.	Copper chloride required. Grm.	Difference. Grm.
Experiment I.	0.1030	0.1069	0.1738	0.1745	-0.0007
" II.	0.1004	0.1077	0.1701	0.1713	-0.0012
" III.	0.1026	0.1060	0.1741	0.1738	+0.0003
" IV.	0.1019	0.1058	0.1718	0.1726	-0.0008

	Bismuth trioxide obtained. Grm.	Bismuth trioxide required. Grm.	Difference. Grm.
Experiment I.	0.1076	0.1069	+0.0007

The time required in each of these trials was seven hours. It seemed to be advantageous to raise the temperature and heat sharply for about ten minutes at the end, to insure the complete removal of the bismuth.

Moistening with acid helped the reaction, but subjected it to the same danger of creeping as noted under antimony and copper.

* From author's thesis presented to the Faculty of the University of Pennsylvania for the degree of Ph.D., 1896. From the *Journ. Amer. Chem. Soc.*, xviii., December, 1896.

The bismuth was estimated as follows: It was washed out of the tube and bulb with acidulated water, and then precipitated as sulphide. The bismuth sulphide was filtered, washed, and dissolved in nitric acid. It was thrown out of the solution with ammonium hydroxide and ammonium carbonate as hydrated oxide, and then filtered, dried, and ignited. It was weighed as oxide. The residue of copper chloride in the boat dissolved in cold water and showed no bismuth.

IX.—Action of Gaseous Hydrochloric Acid on Sodium Pyroarsenate.

Hibbs (*Journ. Amer. Chem. Soc.*, xviii., 1044) showed that arsenic was completely volatilised from sodium pyroarsenate, leaving weighable sodium chloride. In fact, so clean was the elimination of arsenic that he made it the basis of an arsenic atomic mass determination, with admirable success.

In working up the separation of arsenic from other metals it was necessary to start with the pure sodium salt. After purification I decided to test it, by weighing the salt produced by the action of the acid gas upon it. Several determinations gave close results, proving the salt pure.

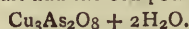
Chemically pure arsenate was procured. It was re-crystallised and then ignited (not too strongly) for an hour. The pyroarsenate obtained was used in precipitating the various arsenates investigated.

	Sodium pyroarsenate taken.	Sodium chloride obtained.	Sodium chloride required.
	Grm.	Grm.	Grm.
Experiment I.	0.2021	0.1330	0.1335
" II.	0.1039	0.0691	0.0686

X.—The Separation of Arsenic from Copper.

Pure sodium pyroarsenate was used to precipitate the copper salt.

Copper sulphate was re-crystallised five times, a few good crystals were dissolved and the two solutions mixed. A green copper arsenate was precipitated. It was washed and dried at 100°. Salkowski (*Journ. Prakt. Chem.*, civ., 129) observes that copper arsenate still contains water above 130°. My salt had the composition—



Hydrochloric acid gas completely changes it in the cold to chloride. A slight heat drives out the arsenic and water and leaves a brown anhydrous copper chloride, which can be weighed as such. Care was taken to remove all the acid gas before weighing.

The arsenic was washed out of the bulb into a beaker; this was warmed with nitric acid to insure oxidation, and then it was precipitated from an ammoniacal solution with "a magnesia mixture." It was weighed as $\text{Mg}_2\text{As}_2\text{O}_7$.

	Copper arsenate taken.	Copper chloride obtained.	Copper chloride required.	Difference.
	Grm.	Grm.	Grm.	Grm.
Experiment I.	0.1067	0.0850	0.0851	-0.0001
" II.	0.1240	0.0998	0.0991	+0.0007
" III.	0.1072	0.0860	0.0856	+0.0004
" IV.	0.1155	0.0924	0.0923	+0.0001
" V.	0.1042	0.0832	0.0833	-0.0001

Experiment I. As_2O_5 obtained, 0.0498 grm.; As_2O_5 required, 0.0487 grm.

The residue of copper chloride completely dissolved in water. It showed no arsenic when tested in a Marsh apparatus.

XI.—The Separation of Arsenic from Silver.

Silver arsenate was made by precipitating silver nitrate with sodium arsenate. Care was taken to have the nitrate in excess. The reddish-brown arsenate of silver was washed with boiling water, until the washings no

longer showed silver when tested with hydrochloric acid. It was dried at 110°.

As was expected, the acid gas attacked it even in the cold. In fact the action was so vigorous that a couple of analyses were spoiled by spattering. The trouble arose from the fact that the arsenate was not finely powdered. Heat was generated in the reaction sufficiently to send over a portion of the water formed. Experiment I. was run in the cold for one hour and then heated sharply for a few minutes to expel the arsenic and water. The result was only 0.46 per cent too high, but indicated that the salt should be heated longer, and not necessarily as high to remove all the arsenic.

The succeeding experiments were heated from one to two hours at 150° with better results:—

	Silver arsenate taken.	Silver chloride obtained.	Silver chloride required.	Difference.
	Grm.	Grm.	Grm.	Grm.
Experiment I.	0.2542	0.2381	0.2363	+0.0018
" II.	0.2325	0.2163	0.2161	+0.0002
" III.	0.2084	0.1952	0.1938	+0.0014
" IV.	0.2070	0.1927	0.1924	+0.0003

Experiment I. Ag obtained = 70.45 per cent; Ag required = 69.99 per cent.

The residues in Experiments II., III., and IV. were dissolved and tested for arsenic. None was found.

XII.—The Separation of Arsenic from Cadmium.

Chemically pure cadmium sulphate was precipitated by a solution of sodium pyroarsenate. Stirring brought out a gelatinous arsenate, which changed by additional stirring to a granular salt. This was thoroughly washed and dried at 110°. It had the composition $\text{Cd}_3\text{As}_2\text{O}_8 + 2\text{H}_2\text{O}$. Salkowski (*loc. cit.*) observes that a red heat is necessary to fully dehydrate this salt.

The moisture and arsenic were completely expelled at 150°, leaving a uniform mass of cadmium chloride. It was weighed as such after standing over sulphuric acid for one-half hour. The arsenic was determined as usual.

	$\text{Cd}_3\text{As}_2\text{O}_8 + 2\text{H}_2\text{O}$ taken.	Cadmium chloride obtained.	Cadmium chloride required.	Difference.
	Grm.	Grm.	Grm.	Grm.
Experiment I.	0.2359	0.1965	0.1977	-0.0012
" II.	0.1166	0.0968	0.0968	0.0000
" III.	0.1030	0.0857	0.0855	+0.0002
" IV.	0.1138	0.0947	0.0946	+0.0001
" V.	0.1043	0.0870	0.0867	+0.0003

	$\text{Cd}_3\text{As}_2\text{O}_8 + 2\text{H}_2\text{O}$ taken.	As_2O_5 obtained.	As_2O_5 required.	Difference.
	Grm.	Grm.	Grm.	Grm.
Experiment I.	0.2359	0.0813	0.0822	0.0009

The cadmium chloride dissolved perfectly in water, and showed no arsenic when tested in a Marsh apparatus.

XIII.—The Action of Hydrochloric Acid Gas on Ferric Oxide.

Pure oxide of iron was heated in a stream of acid gas. The behaviour of iron is rather peculiar, as it very readily changes into chloride, and then only partially volatilises. On heating to 200° the greater part is driven over as flaky crystals of ferric chloride. The remainder consists of a white mass, which refuses to go over on prolonged action and also on raising the temperature.

This residue was soluble in water and did not react with potassium thiocyanate, but immediately gave a blue precipitate with ferricyanide. Reduction was therefore evident; this is also noted by Jannasch and Schmidt (*loc. cit.*). The temperature at which ferric chloride usually goes into the ferrous condition is above 1000°.

Care was taken to prepare perfectly pure hydrochloric acid gas. Chemically pure acids were used to this end. The action, however, was the same in all cases.

XIV.—The Separation of Arsenic from Iron.

Chemically pure ferrous ammonium sulphate was carefully oxidised with nitric acid, it was taken up in water, filtered, and then crystallised several times. The best crystals were selected, and a solution made to precipitate the arsenate. A white precipitate tinged with yellow was formed. It was washed by decantation and then filtered and washed until the washings no longer gave Prussian blue with ferrocyanide. It was then dried and gently ignited.

The acid gas acts on it quickly in the cold, and it becomes a light green liquid. In evaporating off the moisture the chloride of iron was carried over with the arsenic.

In a second trial, with the temperature lower and occasionally removing the source of the heat altogether, when ebullition threatened to cause spattering, ferric chloride was obtained without loss. This was gradually heated a little higher to remove all the arsenic.

The chloride of iron was dissolved, oxidised, precipitated with ammonium hydroxide, and estimated as usual. The result was fair, and the product tested showed the absence of arsenic, but all succeeding experiments failed. Either the substance spattered or the iron went along with the arsenic.

Jannasch and Schmidt (*loc. cit.*) separated arsenic from iron by placing their material in a large hard glass bulb and evaporating down to dryness with nitric acid in an air current. This is not applicable when a porcelain boat is employed. They then volatilised the arsenic in hydrochloric acid gas at 120°.

(To be continued).

NICKELO-NICKELIC HYDRATE, $\text{Ni}_3\text{O}_4 \cdot 2\text{H}_2\text{O}$.

By WILLIAM L. DUDLEY.

IN studying the action of fused sodium dioxide on metals, I have obtained interesting crystalline compounds, some of which at least have never been described. Only one of them has been carefully investigated, and it proves to be nickelo-nickelic hydrate, having the formula $\text{Ni}_3\text{O}_4 \cdot 2\text{H}_2\text{O}$.

It is prepared by fusing sodium dioxide in a nickel crucible with metallic nickel at a cherry-red heat. The action of the oxide upon the nickel proceeds with moderate rapidity, and in a few minutes scaly crystals appear floating in the fused mass. The crystals multiply steadily until, in the course of an hour, the contents of the crucible is thick with them, and comparatively little liquid remains. After cooling, the crucible is submerged in a beaker of distilled water, and the undecomposed sodium dioxide, together with the sodium oxide, dissolves out, leaving the crystals which rapidly settle to the bottom of the liquid. The crystals should be washed several times with boiling water by decantation, and finally thrown in a filter. It is quite difficult to wash out all the alkali, which adheres with unusual persistence. Probably the best plan to adopt is to put the crystals in a Soxhlet extraction apparatus, and wash with water until no colouration is obtained with phenolphthalein. This requires about fifty hours of continuous washing. The crystals should then be dried at 110° C., and a magnet passed carefully through them to remove any particles of metallic nickel which may have eroded and not been completely acted upon.

The crystals are lustrous and almost black, with a slight brown-bronze hue. They are soft, and grind in a mortar much like graphite. The crystals seem to be hexagonal plates, but measurements of the angles have not been made. They dissolve slowly in acids, forming nickelous salts. Hydrochloric acid evolves chlorine; sulphuric and nitric acids, oxygen. They are insoluble in water and in solutions of the alkalis. The compound is not magnetic. The specific gravity is 3.4115 at 32° C.

At 130° C. the compound does not undergo decomposition, but at about 140° C. it begins to lose weight; at 240° C. the weight remains constant. At a red heat further loss is sustained and the residue remaining is nickelous oxide. The loss from 130° C. to 240° C. is due to water driven off, and at a red heat this loss is due to the evolution of oxygen.

The compound proved to be $\text{Ni}_3\text{O}_4 \cdot 2\text{H}_2\text{O}$, as is shown by the results of the analysis:—

	Per cent.
Loss of H_2O on heating from 130° C. to 240° C.:—	
First determination	13.00
Second	13.13
Theory for $\text{Ni}_3\text{O}_4 \cdot 2\text{H}_2\text{O}$	13.06

The residue remaining after heating to 240° C. is Ni_3O_4 . On heating this residue to redness the loss of oxygen was found to be:—

	Per cent.
Loss of oxygen	6.63
Theory	6.67

The total loss of water and oxygen obtained on heating the compound from 130° C. to redness was:—

	Per cent.
First determination	18.91
Second	18.88
Theory for $\text{Ni}_3\text{O}_4 \cdot 2\text{H}_2\text{O}$	18.86

The oxygen given off on heating to redness was determined by calcining the compound in an atmosphere of carbon dioxide, and collecting in Schiff's apparatus over potassium hydroxide solution. The result gave:—

	Per cent.
Oxygen	5.93
Theory for $\text{Ni}_3\text{O}_4 \cdot 2\text{H}_2\text{O}$	5.84

The nickel was determined and found to be:—

	Per cent.
Nickel	63.67
Theory	63.72

In all of the calculations the atomic weight of nickel was taken to be 58.56, and oxygen 16.

The compound made in a nickel crucible of commerce is not perfectly pure, as the sample obtained was found to contain 0.71 per cent of cobalt, the presence of which, however, would make no appreciable difference in the results of the analyses. No method has been found for freeing the compound from this impurity, and it appears at present as if the only plan would be to use a chemically pure nickel crucible in making it, for no crucible will withstand the action of fused sodium dioxide. Porcelain, iron, silver, gold, and platinum crucibles are rapidly attacked.

The presence of water in this compound seems curious, but it may be due to the presence of sodium hydroxide in the sodium dioxide. Again it may be due to the water added to dissolve the soluble residue from the crystals. The first explanation seems to be the more plausible since the crystals are formed in the mass while it is fused, and they are not produced upon the addition of the water. If such is the case it would seem that the water driven off between 130° C. and 240° C. is from the breaking down of a true hydrate, rather than the expulsion of water of crystallisation.

A cobalto-cobaltic hydrate, $\text{Co}_3\text{O}_4 \cdot 2\text{H}_2\text{O}$, has been described (Genth and Gibbs, *Amer. Journ. Sci.*, xxiii., 257), but it was obtained by exposing to moist air, Co_3O_4 , prepared by heating cobalt carbonate. Ni_3O_4 , prepared by heating nickelo-nickelic hydrate to 240° C., is hygroscopic, and absorbs about 7.16 per cent of water from the air at 30° C., which is completely lost at 110° C., showing that no hydrate is formed under these conditions.

The study of the action of fused sodium dioxide on the metals will be continued here, and it is hoped that some more data can be contributed soon.—*Journal of the American Chemical Society*, xviii., October, 1896.

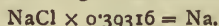
ALUMINUM ANALYSIS.*

By JAMES OTIS HANDY.

(Continued from p. 56).

Determination of Sodium in Aluminum.

ONE grm. of drillings is dissolved in a porcelain evaporating dish in 50 c.c. of 1.3 sp. gr. nitric acid and sufficient hydrochloric acid to effect solution. Boil down until all hydrochloric acid has been removed. Rinse the solution into a large platinum dish and evaporate to dryness. Heat over a good Bunsen burner until nitric oxide fumes cease to be evolved. Grind the residue finely. Mix it by grinding with 1 grm. of chemically pure ammonium chloride and 8 grms. of chemically pure calcium carbonate. Heat the mixture in a large covered platinum crucible. For the first fifteen minutes have a Bunsen burner flame just touching the bottom of the crucible, and for the next forty-five minutes have the whole crucible heated bright red by a full Bunsen burner flame. Cool, and treat the residue with hot distilled water until it becomes just friable under pressure. Avoid adding an excess of water beyond that necessary to make the sintered mass just friable. Grind it in a Wedgwood mortar, and rinse out with hot distilled water. Filter, rejecting the well-washed residue, and treat the filtrate at the room temperature with saturated ammonium carbonate solution in slight excess. Stir very thoroughly. The precipitated calcium carbonate is at first flocculent, but on standing for about ten minutes it becomes crystalline. Filter into a platinum dish; reject the residue, and evaporate the solution on the water-bath to dryness. Heat carefully to dull redness to expel ammonium salts. Dissolve the residue in a little water, and add a few drops of ammonium carbonate solution. If this produces a precipitate, add sufficient ammonium carbonate solution to precipitate all of the remaining lime. Stir well, wait ten minutes, filter, evaporate to dryness, heat to dull redness, and weigh sodium chloride. Deduct any sodium chloride found in a blank determination, using acids, &c., as above, and finally 8 grms. of calcium carbonate and 1 grm. of ammonium chloride.



Care should be taken when heating up the residue of sodium chloride, &c., after evaporating on the water-bath. If the platinum dish and contents are heated for a few minutes on sheet asbestos on the hot plate before placing over the lamp, spattering may be avoided. Sodium is generally absent from aluminum, but it has been found in amounts as high as 0.20 per cent, and is considered a cause of the occasional deterioration of the metal in water.

Determination of Carbon in Aluminum. (Moissan's Method Modified).

TRITURATE 2 grms. of drillings in a Wedgwood mortar with 10 to 15 grms. of mercuric chloride, powdered and dissolved, or partly dissolved, in about 15 c.c. of water. Reaction takes place rapidly, and a heavy grey residue is left. Persistent trituration removes the last particles of metallic aluminum. Evaporate on the water-bath to dryness. The dry residue is heated in a current of pure hydrogen to expel mercuric compounds. The remaining material is then placed in a boat in a combustion-tube and burned off as in carbon determination in steel. The carbon dioxide is caught as barium carbonate, and the excess of barium hydroxide determined by means of standard acid. We are working on a more generally applicable method for carbon in aluminum.

Determination of Nitrogen in Aluminum.

ALUMINUM, when overheated in re-melting, is believed to have the property of combining with nitrogen. The metal becomes weaker. Moissan's method for deter-

mining nitrogen in aluminum may be found in *Comptes Rendus*, cxix., 12. Nitrogen thus absorbed would undoubtedly exist as nitride of aluminum, and solution of sodium hydroxide with subsequent distillation would seem to be the best method of procedure. We are working up this method.

Determination of Aluminum in Metallic Aluminum.

Dissolve 1 grm. of metal in 30 c.c. of 33 per cent hydrochloric acid in a porcelain dish and evaporate cautiously to complete dryness. Re-dissolve, by boiling with 10 c.c. of concentrated hydrochloric acid and 75 c.c. of water. Wash into a 12-ounce beaker; dilute to 250 c.c., and pass hydrogen sulphide until saturated. Filter into a beaker and boil off hydrogen sulphide. Oxidise by adding 1 c.c. of concentrated nitric acid and continuing to boil for ten minutes. Cool, and make the solution up to 500 c.c. Remove 50 c.c. of the solution, and, having diluted to 250 c.c. and heated to boiling, add ammonium hydroxide in slight excess and boil well for twenty minutes. Let settle; filter, and wash thoroughly with hot water. It is necessary to wash the precipitate off from the filter, break it up, and wash it back again. Finally burn off in a thin-walled platinum crucible, igniting most intensely, and weighing the instant the crucible and contents are cool. We have found that alumina is one of the most difficult oxides to dehydrate completely, and when dehydrated it absorbs atmospheric moisture even more rapidly than calcium oxide does. Moissan prefers to precipitate aluminum by ammonium sulphide. Having prepared a solution in hydrochloric acid, he takes an amount equal to 0.15 grm. of aluminum, neutralises it in the cold with ammonia, and precipitates it by ammonium sulphide which has been recently prepared. He then digests for one hour, filters, washes with hot water, ignites, and weighs.

Analysis of Alloys of Aluminum with Smaller Amounts of other Metals.

Copper Alloys.—Three to thirty per cent copper, and no zinc or nickel.

Dissolve $\frac{1}{2}$ grm. or 1 grm. in 15 c.c. of 33 per cent sodium hydroxide solution in an Erlenmeyer flask of 12-ounce capacity. If the flask is covered and set in a warm place, solution is complete in a few minutes, even if the drillings are quite coarse. Dilute to 30 c.c. with hot water, and filter through a coarse lintless filter-paper (Whitall, Tatum, and Co.'s 5-inch). Wash well with hot water. Dissolve residue, after washing it off the filter-paper into a 12-ounce beaker, by warming with 5 c.c. of concentrated nitric acid. Cool, add saturated commercial sodium carbonate solution until re-solution occurs. Titrate with standard potassium cyanide solution to the disappearance of the blue colour. Standardise the cyanide for about the same amount of copper.

For commercial reasons 20 per cent alloys are made in the reduction pots, and these alloys are subsequently used for making copper alloys of low percentage.

Determination of Nickel in Aluminum Alloys.

The 3 per cent nickel alloy is now used. The addition of 3 per cent of nickel increases the tensile strength of aluminum by several thousand pounds per square inch.

One grm. of drillings is dissolved in 15 c.c. of 33 per cent sodium hydroxide solution in a 12-ounce Erlenmeyer flask. Dilute to 50 c.c., and filter through a 5-inch lintless paper, washing the residue thoroughly with hot water. Rinse the residue back into the flask, and add 3 to 5 c.c. of concentrated nitric acid, and a drop of concentrated hydrochloric acid. Boil, and when dissolved cool, and make up to 250 c.c. In 100 c.c. determine the copper by neutralising with ammonia, adding 2 c.c. of concentrated hydrochloric acid, warming, and passing hydrogen sulphide. Filter and wash with ammonium sulphide. Burn it off carefully in a porcelain crucible, and, having weighed, dissolve in 5 c.c. of concentrated nitric acid.

* From the *Journal of the American Chemical Society*, Sept., 1896.

Then dilute to 20 c.c., add excess of sodium carbonate solution, and titrate with standard potassium cyanide. Boil the filtrate from the cupric sulphide, oxidise with 1 c.c. of nitric acid, and precipitate with ammonium hydroxide. Do not boil, but digest for a few minutes just below the boiling-point. Filter, wash, re-dissolve in hot 15 per cent nitric acid wash. Dilute to 150 c.c., and again precipitate with excess of ammonium hydroxide, being careful to avoid boiling or prolonged digestion. Filter and wash. Burn off and weigh ferric oxide, &c. In a second 100 c.c. of the main solution, precipitate nickel hydroxide, cupric oxide, ferric hydroxide, &c., by 33 per cent chemically pure sodium hydroxide solution, added in slight excess to the boiling solution. Boil for fifteen minutes, filter, and wash most thoroughly with hot water. Burn off and weigh nickel oxide, cupric oxide, and ferric oxide. Deduct cupric oxide and ferric oxide already found. Calculate nickel oxide to metallic nickel.

Analysis of Aluminum-Manganese Alloys.

Determination of Manganese.—Place 1 gm. of drillings in a 12-ounce beaker. Add 30 c.c. of 33 per cent hydrochloric acid (one part of concentrated hydrochloric acid to two parts of water). When dissolved add 25 c.c. of nitric acid (1·42), and boil down to 10 c.c. Add 50 c.c. of colourless nitric acid (1·42), and boil. Precipitate the manganese with powdered potassium chlorate, added cautiously, and proceed as described under manganese in steel by Williams's method (Blair's "Chemical Analysis of Iron").

Analysis of Chromium-Aluminum Alloy.

Determination of Chromium.—Dissolve 1 gm. in a 12-ounce beaker in 30 c.c. of 33 per cent hydrochloric acid, and when dissolved add 50 c.c. of sulphuric acid (1·84), and evaporate carefully until fumes of sulphur trioxide escape. Cool, add 60 c.c. of water, and boil. After five minutes, if all aluminum sulphate has been dissolved, add powdered potassium permanganate until the solution has a distinct pink colour. Boil until the excess of potassium permanganate is decomposed. Filter through washed asbestos, and determine the chromium in the filtrate as in chrome steel (Galbraith's method. See Blair's "Chemical Analysis of Iron").

Analysis of Tungsten-Aluminum Alloy.

Determination of Tungsten.—Dissolve 1 gm. in 33 per cent hydrochloric acid in a 4½-inch evaporating dish. Add 30 c.c. of nitric acid (1·42), and evaporate to dryness. Re-dissolve in 30 c.c. of hydrochloric acid (1·20), dilute to about 90 c.c., and boil for two hours. Filter and wash thoroughly. Burn off and weigh $\text{Si} + \text{SiO}_2 + \text{WO}_3$ + crucible. Treat with 3 drops of 25 per cent sulphuric acid and about 2 c.c. of hydrochloric acid. Evaporate carefully over an Argand burner, re-ignite, and weigh crucible and silicon and tungstic oxide. Fuse with 1 gm. of sodium carbonate, cool, place in dish, and add 15 c.c. of water and 20 c.c. of 25 per cent sulphuric acid, remove crucible, and evaporate until white fumes escape. Cool, re-dissolve in about 50 c.c. of water. Filter, wash, ignite, and weigh silica (from silicon), tungstic oxide, and crucible. Treat with sulphuric acid and hydrofluoric acid, evaporate, ignite, and re-weigh. Loss equals silica. Calculate to silicon, and add to the weight of silica lost by treatment of first insoluble residue. Deduct this sum from the weight of silicon, silica, and tungstic oxide first found, and the remainder equals tungstic oxide. Calculate to tungsten.

Analysis of Aluminum-Titanium Alloy.

Determination of Titanium.—Two grms. of the alloy in a 12-ounce Erlenmeyer flask are dissolved by addition of 50 c.c. of 10 per cent potash solution. Dilute with distilled water to about 125 c.c., boil up, and filter as quickly as possible. Wash ten times with boiling water.

Burn off the residue in a porcelain crucible, crush it in a Wedgwood mortar, fuse in a large platinum crucible with 10 grms. of potassium bisulphate. Conduct the fusion exactly as follows:—Choose a good Bunsen burner, and protect it from draught by a sheet-iron chimney; make the flame 1½ inches long, and place the triangle carrying the upright crucible just at the point of the flame. Increase the heat gradually until in ten minutes the lower fourth of the crucible is red hot. Allow it to remain at this temperature ten minutes, moving the lid slightly to one side every two minutes, and giving the crucible (held firmly in the tongs) a gentle rotating movement, then turn up the light until the flame reaches the top of the crucible and envelopes it. Five minutes of this treatment melts down any potassium bisulphate, &c., which have risen on the sides. The flame is lowered and the lower fourth heated for ten minutes longer. Cool, dissolve in about 200 c.c. of water; filter, rejecting the residue, if ignition and treatment with hydrofluoric acid show it to be only silica. If it contains anything more, fuse with 4 grms. of potassium bisulphate again. The filtrate contains all the titanic oxide and the ferric oxide. Add ammonia until a slight permanent precipitate is formed, then add dilute sulphuric acid from a pipette or burette until this precipitate just re-dissolves. Finally add 1 c.c. more of 25 per cent sulphuric acid, and dilute to 300 c.c. If the solution is high in iron (which will be indicated by its distinct yellow colour) sulphur dioxide gas must be run into it until it is decolourised and smells strongly of sulphur dioxide; but if the solution is nearly colourless, indicating a low percentage of iron, only sulphur dioxide water need be added for the reduction. Boil well for one hour, adding water saturated with sulphur dioxide occasionally. Filter off the titanic oxide through double filters, and wash well with hot water. Burn off and weigh as titanic oxide. If the precipitate is yellow, indicating the presence of iron, it may be fused with 1 gm. of potassium bisulphate, the fusion dissolved in 10 c.c. of dilute sulphuric acid, and the iron determined in this solution by reducing with 1 gm. of zinc, and titrating with permanganate. This is not often necessary. Calculate titanic oxide to titanium. $\text{TiO}_2 \times 0.6 = \text{Ti}$.

Determination of Zinc in Zinc-Aluminum Alloys. First Method.

Dissolve 1 gm. in 30 c.c. of 33 per cent hydrochloric acid in a 12-ounce beaker. Dilute to 200 c.c., and heat nearly to boiling. Pass hydrogen sulphide till all copper is precipitated. Filter and boil off hydrogen sulphide, oxidise with 1 c.c. nitric acid by boiling ten minutes. Add sodium hydroxide solution until neutral, then make barely acid with hydrochloric acid, and stir until the aluminum hydroxide all dissolves. Add 10 grms. of sodium acetate and 500 c.c. of water, boil up, and filter at once. Dissolve the washed precipitate in hydrochloric acid, and repeat the acetate separation. Heat the united filtrates to boiling and pass hydrogen sulphide. Filter off the zinc sulphide on double filters, wash thoroughly with hot water. Burn off in a porcelain crucible, and weigh zinc oxide. Calculate to zinc. This method may be used when only a small quantity of the sample is available; but when this is not the case, it is better to use the method given below.

Determination of Zinc in Zinc-Aluminum Alloy. Second Method.

Dissolve 1 gm. of drillings in 33 per cent sodium hydroxide solution in a 12-ounce Erlenmeyer flask. Filter as soon as dissolved through a 4-inch lintless filter-paper. Wash thoroughly with hot water. Rinse the residue of zinc, iron, copper, silicon, &c., back into the flask. This may require 25 c.c. of water. Add 5 c.c. of hydrochloric acid and boil. Dilute to 150 c.c. with hot water and pass hydrogen sulphide. Filter and boil off hydrogen sulphide, re-oxidise by adding 1 c.c. nitric acid and boiling ten minutes. Add sodium hydroxide till neutral, then add

dilute hydrochloric acid till just acid, and then 10 grms. of sodium acetate, and 300 c.c. of boiling water, and boil for five minutes. Wash well. If the precipitate is small re-solution and re-precipitation are not necessary. Pass hydrogen sulphide through the filtrate. Filter off zinc sulphide through double filters. Wash well. Ignite in a porcelain crucible, heating finally over the blast to zinc oxide. $\text{ZnO} \times 0.832 = \text{Zn}$.

Analysis of Aluminum Solders.

Determination of Tin, Phosphorus, and Zinc.—Aluminum solders generally contain phosphor-tin and zinc. As presented for analysis they usually consist of a soldered joint, from which the solder must be scraped and analysed. The analysis, therefore, involves a separation of the elements aluminum, zinc, tin, and phosphorus. It is a difficult matter to determine whether aluminum was a constituent of the solder when only a soldered joint is available for examination. It is best to dissolve all adhering aluminum from the pieces chosen for analysis by treatment with 33 per cent sodium hydroxide solution, after which the residue is filtered off, dried, and weighed out for analysis. Dissolve or decompose three-tenths to five-tenths grm. in a twelve-ounce beaker by means of 20 c.c. of nitric acid (1.42). If necessary, 5 c.c. of hydrochloric acid (1.2) may be used to effect complete decomposition. Evaporate to complete dryness on a hot plate. Cool, add 25 c.c. of nitric acid (1.13), and boil thoroughly. Filter. The residue contains all of the tin, most of the phosphorus, and possibly some zinc. Burn it off in a porcelain crucible, and, after pulverising the residue in an agate mortar, mix it with 2 grms. of sodium carbonate and 2 grms. of sulphur, fuse it in a covered porcelain crucible over a Bunsen burner for about half-an-hour. Give it three minutes of gentle blast flame at the last. Cool, boil out with 150 c.c. of water in a twelve ounce covered beaker. Filter and wash. Extract any possible zinc sulphide, &c., from the residue by dissolving in nitric acid, boiling off hydrogen sulphide, and adding this to the first filtrate obtained after evaporating to dryness with nitric acid. The sodium sulphide solution contains the tin and phosphorus. Add it to hydrochloric acid until just acid. Warm slightly and pass hydrogen sulphide. Filter off stannous sulphide and wash thoroughly with hot water. Burn off in a porcelain crucible and weigh stannic oxide. Calculate to metallic tin. The filtrate from the stannous sulphide is boiled to expel hydrogen sulphide, and then oxidised by adding 2 c.c. of nitric acid and boiling for fifteen minutes more. Filter off any sulphur which separates, and in this filtrate, which should amount to only about 100 c.c., precipitate the phosphorus by adding pure sodium hydroxide solution till alkaline, then nitric acid till distinctly acid, heating to 85° C., and adding 50 c.c. of filtered molybdate solution. Stir or shake well for five minutes, filter on a weighed filter paper, and after washing with one per cent nitric acid wash, dry at 100° C., and weigh. Yellow precipitate multiplied by 0.0163 equals phosphorus. The nitric acid solution obtained after evaporating the first solution to dryness, &c., is now neutralised with sodium hydroxide solution, and then made just acid with hydrochloric acid. Ten grms. of sodium acetate are now added, and 300 c.c. of water (hot). Boil up for five minutes, then filter and wash. If the precipitate is of considerable size, it is probable that aluminum was a constituent of the solder. Re-dissolve it in a little hydrochloric acid, neutralise, acidify, and make a basic acetate separation as before. Precipitate the zinc in the acetate solutions by hydrogen sulphide. Filter, wash, ignite in a porcelain crucible, and weigh as zinc oxide. Calculate to metallic zinc. Dissolve the precipitate of aluminum acetate in hydrochloric acid, dilute to 250 c.c., and precipitate with ammonia. After filtering, washing, igniting, and weighing as alumina, calculate to metallic aluminum. Solders containing lead are sometimes met with. In such cases, evaporate the nitric acid filtrate from the metastannic

acid to small bulk, add 25 c.c. of 25 per cent sulphuric acid, and evaporate until white fumes escape. Cool, add 100 c.c. of water, stir, and let stand for an hour in a warm place. Filter and wash with water containing 5 per cent sulphuric acid. Burn off in a porcelain crucible at a low temperature. Re-oxidise any reduced lead oxide, and restore its sulphur trioxide by adding a few drops of nitric acid and sulphuric acid and evaporating. Finally weigh lead sulphate. Calculate to metallic lead. Zinc is determined in the lead sulphate filtrate.

Analysis of Alumina.

Alumina is made from bauxite or cryolite. It is usually purchased in the hydrated form.

(To be continued).

PROCEEDINGS OF SOCIETIES.

EDINBURGH UNIVERSITY CHEMICAL SOCIETY.

At a meeting of the Edinburgh University Chemical Society on the 11th January, a paper was read by Dr. Macdonald on the "*Constitution of Benzene*."

The different formulæ from time to time proposed for benzene were discussed and criticised. It was pointed out that for the study of all ordinary benzene derivatives, Kekulé's hexagonal formula had proved the most serviceable. The identity of 3- and 5-methylpyrazole is much akin to the benzene tautomerism, and points more clearly to re-arrangement of single and double bonds such as proposed in Kekulé's oscillation hypothesis. This hypothesis is difficult to accept in detail, but for a working hypothesis it is sufficient to assume that in certain peculiar conditions of ring symmetry double linkings are not fixed. The disturbing agent might be found in the intra-molecular energy. Assuming this to take the form of energy of motion round the ring, the single and double bonds would then represent something like the phases of a wave motion.

At the Fourth Ordinary Meeting, held on Monday, Jan. 25th, 1897 (Dr. Macdonald in the chair), Dr. DOBBIN read a paper on the subject—"Who Introduced the Use of the Balance into Chemistry?"

After quoting a variety of statements from current textbooks, which more or less emphatically attributed to Lavoisier the discovery of the law of the conservation of matter, and the first employment of the balance in investigating theoretical questions in chemistry, the author of the paper remarked upon the startling character of these statements to any person who had read Black's research on "Magnesia Alba," and quoted a few passages from Black in support of the statement that every step in his investigation has been made good by appeal to direct quantitative experiments. He next quoted a passage from "La Révolution Chimique," showing that Berthelot was well aware of the fact that these views regarding Lavoisier were entirely at variance with the true state of matters.

Dr. Dobbin then went on to mention that, so far as he was aware, the well known and often quoted experiment of van Helmont upon the supposed formation, from water only, of 164 pounds weight of the substance of a willow tree—the weight of the earth in which this willow grew having varied only by about two ounces in five years—was the earliest attempt to determine the accuracy of a view concerning a matter of scientific fact by appeal to quantitative experiment. It was further pointed out that Boyle made very frequent use of the quantitative method of investigation in dealing with the most diverse subjects, and that his inspiration in this direction was very probably derived from van Helmont.

The author concluded his paper by pointing out, with regard to Lavoisier's examination of the alleged conversion of water into earth, that every essential point in the investigation, and in the mode of carrying it out, was to be found discussed or suggested in the works of Boyle, of which we know that Lavoisier was an attentive student. So far as he had been able to ascertain, attention had not previously been called to this fact by the historians of chemistry.

NOTICES OF BOOKS.

The Manufacture and Properties of Structural Steel.
By HARRY HUME CAMPBELL, S.B. New York and London: The Scientific Publishing Co. 8vo., pp. 397. 1896.

If we were called upon to classify this valuable work we should feel at a loss. A chemical treatise it certainly is not, seeing that the metal which it discusses is characterised not by its chemical composition, qualitative or quantitative, and is hence named "structural steel." Chemical considerations are certainly not overlooked, but they play here a relatively less weighty part than they do, e.g., in tissue-printing or in alkali-making. If we bear in mind that steel and iron works are in the hands of engineers, and that the authorities here quoted are the proceedings of societies for civil and mechanical engineering we must place this work under the category of "engineering."

The author in his first chapter brings forward the "errancy" of scientific records—a painful subject under a strange name. Errors are shown in Howe's "Metallurgy of Steel."

These variations in the results of experiments are due to the accumulation of petty errors. It is of some consolation to find that the variations here cited are not in chemical composition, but in physical properties, such as ultimate strength, elastic limit, percentage of elongation, reduction of area, and percentage of elastic ratio. But variations in chemical composition are also quoted. The different methods of determining carbon vary to an extent unexpected as unsatisfactory.

The author remarks—as it might be fairly expected—that the comparison of miscellaneous records is perfectly useless and misleading. "Even the results of two different well-conducted laboratories may not be trustingly placed together. This may be done if the two works in question exchange samples and find that both obtain similar results from the same metals, but under no other circumstances is the comparison valid."

In the determination of phosphorus similar but more important errors are committed.

The author concludes this chapter with the pithy dictum: "The making of steel was once a trick; it was then an art; it is now a business."

Chapter IV. leans to the definition of steel, a task which though apparently simple has never yet been accomplished to the satisfaction of all concerned. "A true formula," says an author, "must apply not only to all the metals commonly designated by the term, but to all compounds which ever have been or ever will be worthy of the name, including the special alloys made by the use of chromium, tungsten, nickel, and other elements introduced to give peculiar qualities for special purposes."

After criticising various proposed definitions the author gives the following standards as embodying current usage in America, and become universal also in Britain and in France. In other lands the authority of famous names, backed by conservatism (word wrongly used!) governmental prerogative, has fixed for the present in metallurgical literature a list of terms which I have tried to show is not only difficult but fundamentally false.

The author understands therefore by the term wrought

iron the product of the puddling furnace or the sinking fire. On the other hand, "by the term steel is meant the produce of the cementation process, or the malleable compounds of iron made in the crucible, the converter, or the open-hearth furnace."

In subsequent chapters we have accounts of the acid and the basic Bessemer processes, as also the open-hearth process in its acid and basic modifications.

In Chapter XVII. follows an investigation of the influence of certain elements on the physical properties of steel, the pre-eminence in injurious effects being ascribed to phosphorus. "Safety increases as phosphorus decreases," and the engineer may calculate just how much he is willing to pay for greater protection from accident.

A relatively high proportion of copper has in certain experiments given a slightly higher elastic ratio and a better elongation and reduction of area. These results are scarcely to be viewed as conclusive.

Aluminium has little effect upon tensile strength, while it does not injure the ductility in proportions under 2 per cent.

Notes on the Qualitative Analysis arranged for the Use of Students of the Rensselaer Polytechnic Institute. By W. P. MASON, Professor of Chemistry. Third Edition. Easton, Pennsylvania, Chemical Publishing Co. 1896. 12mo., pp. 56.

In the Preface to this little book we find the admission that "the market is unquestionably much overstocked with books upon this subject," and the author puts forward as his only excuse that it meets the requirements of his own classes. He further expresses the opinion that, were it not for the "expense of printing, every teacher of chemistry would use a text-book made by himself with either pen or scissors." Sad, indeed, if true! He hopes that those who use the matter here given may be led so to think for themselves as to "create a desire to know rather than an anxiety to pass." This is a very laudable aspiration, but we doubt if there is anything calculated to create this desire in this book rather than in not a few others. In the recognition of certain elements the author makes use of the spectroscope and of blowpipe reactions.

An Introduction to the Study of Chemistry. By W. H. PERKIN, Jun., Ph.D., F.R.S., Professor of Organic Chemistry in the Owens College, Manchester; and BEVAN LEAN, D.Sc., B.A., Assistant Lecturer and Demonstrator, and late Berkely Fellow of the Owens College, Manchester. London: Macmillan and Co., Ltd. New York: The Macmillan Co. 1896. Pp. 399.

THIS is a gratifying book. The student is trained to re-discover, or, as the case may be, to re-demonstrate for himself, the cardinal factor and laws of chemical science. Hence, instead of—as is too generally the case—closing the book with the sad reflection that the contents, how true soever, are a mere *rechauffée* of what has been many times said before, we are in a position to congratulate the authors and still more their pupils and readers. They are reminded in the Preface, according to a quotation from Prof. H. A. Miers, that "the order in which problems have presented themselves to successive generations is the order in which they may be most naturally presented to the individual."

In carrying out this plan the author gives an abstract of the birth of chemistry drawn from the invaluable work of Berthelot. The passages cited fully support the maxim that conclusions must be tested by experiment and by measurement. Virgil's strange recipe for generating a swarm of bees from the putrid carcase of a bullock may be traced to the inability of the classical world to distinguish bees from certain carrion-hunting Diptera.

In the chapter on the metric system the authors do not overlook its occasional inconveniences, e.g., its unsuitableness to retail trade. They do not mention the pedantic refusal of French and other instrument makers to admit of any weights except the multiples and submultiples of 10. Had they been willing, instead of the French arrangement of 5, 5, 2, 2, 1, to adopt the English scale, 6, 3, 2, 1, they would have found the metric system gain in popularity.

We are most favourably impressed with Messrs. Perkin and Lean's book, and hope that it may be widely appreciated.

The Progress of Medical Chemistry, comprising its Application to Physiology, Pathology, and the Practice of Medicine. By J. L. W. THUDICHUM, M.D., F.R.C.S.L. London: Baillière, Tindall, and Cox. 1896. Pp. 212.

WE have here a handy book, which will prove of great value to the earnest student of animal chemistry, in perhaps its most complicated region. The character of the work is decidedly controversial, and it possesses a not unpleasant acidulous flavour.

The controversy is mainly directed against the shade of the late Professor F. Hoppe-Seyler, Brücke, E. Salkowsky, Kossel, Freitag, &c. We must not, however, suppose that Dr. Thudichum's hand and pen are raised against all his contemporaries engaged in the same class of researches.

The author commences his work by comments on the "Rise of Specialism, Limited." In a succeeding paragraph he enlarges on the reign of the phagocyte, and the astonishing feats of this creature of the bacteriological imagination, "among which none was more surprising than that by which it put all chemistry to shame—namely, chemotaxis."

We have an elaborate discussion of the protagonist question, which we hope after all its changing phases may now be considered as definitively settled. Dr. Thudichum tells us that the effect of the analytical operations of Kossel and Freitag is entirely retrograde, as they "carelessly repeat indecisive data, ignore facts which have been proved for many years, iterate refuted errors, try to displace proved facts, substitute erroneous names and dates for true ones, and in the only part of their operation which might have furnished something original and new fail in a manner which is the necessary result of disregard for the accepted principles of scientific research."

Towards the end of the book we have a very interesting chapter on "Shady Side of Biological Science," otherwise the ghosts of spurious researches. Following the example of Babbage, he divides spurious researches into four groups—forgeries, hoaxes, cooked and trimmed results.

In addition come the blunderings, made not necessarily in bad faith.

We may thank Dr. Thudichum for some innovations in nomenclature which are decided improvements. For alcohols in its generic sense he would say "cohols." "Quantation" is a convenient abbreviation for quantitative determination.

Dimorphism of the Succinates of the Camphols + α and - α ; Isomorphism of the Succinates of the Camphols + α and - α , and of the Succinates of the Isocamphols + β and - β .—J. Minguin.—As the crystalline form of the succinates approximates decisively to that of borneol and that of camphor, it should seem that a transformation of CO into the CHOR of camphor does not sensibly affect the crystalline form, whilst a transformation effected in CH₂ makes itself more felt.—*Comptes Rendus*, cxxiv., No. 2.

CORRESPONDENCE.

ON THE RELATION OF THE SPECIFIC ROTATORY AND CUPRIC REDUCING POWERS OF THE PRODUCTS OF STARCH HYDROLYSIS BY DIATASE.

To the Editor of the Chemical News.

SIR,—The paper by Messrs. Brown, Morris, and Millar, read before the Chemical Society on December 17th, 1896, and subsequently abstracted and appearing in the *CHEMICAL NEWS*, lxxiv., 43, confirms my own experience in starch estimation in various products by the O'Sullivan process. I have found that, without exception, there is a constant relation between the cupric reducing power and the specific rotatory power, and am, therefore, glad to see a confirmation of my own work by such eminent chemists as the authors of the above paper.—I am, &c.,
J. ARTHUR WILSON.

Newchurch, Rossendale,
Jan. 27, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Zeitschrift für Analytische Chemie.
Vol. xxxvi., Part I.

A Contribution to the Chemistry of Animal Fats.—Carl Amthor and Julius Zinc.—An elaborate paper, not admitting of abstraction.

Determination of Formaldehyd.—Dr. G. Romijn.—For the examination of pure solutions of formaldehyd the iodometric method is to be preferred, on account of its great accuracy and convenient execution. The methods of Brochet and Cambier and the potassium cyanide method may also be recommended. But if the presence of other aldehyds is to be feared the potassium cyanide method is suitable, and, along with the iodometric method, will in many cases permit of the entire analysis of the mixture. Legler's method will never bear a comparison with the other three methods.

Behaviour of the Shellac Acids in the Separation of Fatty Acids and Resin Acids, according to Gladding and Twitchell.—F. Ulzer and Rudolf Defris.—It appears that samples are sold under the name of shellac which consist of a mixture of shellac and ordinary colophonium.

Determination of Thoria in Thorin.—E. Hintz and H. Weber.—This memoir will be inserted in full.

Determination of Fatty Matter in Milk.—H. Fresenius.—The author evaporates the sample over purified quartz-sand, and extracts the dry residue with ether in a suitable apparatus. After distilling off the ether, the residual fat is dried for one hour in a weighing-glass at 100°, and weighed.

A Simple and Universally Applicable Method for Determining the Water in Silicates.—P. Jannasch and P. Weingarten (*Zeit. Anorg. Chem.*).—This paper requires the accompanying illustration.

A Technical Pyrometer.—W. C. Heräus and Keiser and Schmidt.—This instrument depends on the same principle as that of Le Chatelier, and is described in the *Zeit. für Instrumentenkunde*.

Determination of Arsenic.—R. Engel and J. Bernard.—From the *Comptes Rendus*.

Production of Chlorine for Laboratory Purposes.—F. A. Gooch and D. A. Kreider (*Zeit. für Anorg. Chemie*).

Analysis of Purified Zinc.—F. Mylius and O. Fromm (*Zeit. für Anorg. Chemie*).—This memoir will be inserted in full.

Quantitative Determination and Separation of Copper.—F. Mawrow and W. Muttman (*Zeit. für Anorg. Chemie*).—This memoir will be inserted in *extenso*.

Detection of Formaldehyd.—G. Romijn (*Nederland Tijdschrift voor Pharm. Chem. en Toxicologie*).—The author is of opinion that formaldehyd may best be recognised by conversion into hexamethylamin by means of ammonia. At ordinary temperatures this transformation is effected in eighteen to twenty hours. At higher temperatures the conversion is very rapid. The addition of formaldehyd to ammoniacal liquids as a disinfectant is therefore of little value. Romijn gives a number of reactions for the identification of hexamethylamin.

Determination of Nitrogen in presence of Nitrates.—H. C. Sherman.—From the *Journal of the American Chemical Society*.

Execution of the Gravimetric Determination of reducing Sugar by means of Fehling's Solution.—Abstracts are here given of recent papers by Nihoul, Grünhut, Killing, Prager, Farnsteiner, and Hefelmann.

Occurrence and Determination of Copper in Organic Substances, especially in Foods.—From the *Archiv für Hygiene, Chemiker Zeitung, and Kaiserlich Gesundheits amte*.

Normal Constituents of Beerwort which may be supposed to be Abnormal.—J. Brand (*Zeit. für Brauwesen und Dingler's Polyt. Journal, also Berichte*, xxvii., 3115).—Of these substances the principal is maltol, $C_6H_6O_3$. It is probably a methyl-pyroneconic acid.

Examination of Mace.—E. Spaeth.—The fat of genuine mace is yellowish brown, while that of Bombay mace is light yellow.

Volumetric Determination of Lead.—A. C. Beebe. From the *CHEMICAL NEWS*.

Determination of Dry Matter in Peat.—H. Puchnor (*Lonw. Versuchstationen*).—Not suitable for abridgment.

Behaviour of Stannous Chloride with Essential Oils.—E. Hirschsohn.—The author has previously observed that the ethereal oil obtained from Gurgun balsam by means of watery vapour, if boiled with stannous chloride, gives a red colour passing into violet and blue. He proposes examining a series of ethereal oils by this reaction.—(*Pharm. Zeitung Russland*).

Alkaloidal Stearates and their Therapeutic Application.—F. Zonandi.—The author has examined the preparation and properties of the morphin, atropin, and cocain stearates.

Action of Morphine and of Acetanilide upon Mixtures of Ferric Salts and Potassium Ferricyanide.—E. Schaer (*Archiv der Pharmacie*).—This paper is more of theoretical than analytical importance.

Detection and Determination of Sodium Salicylate in presence of Ichthyol (*Journ. de Pharmacie*).—Vissen dries the mixture with fine sand on the water-bath, and extracts in the Soxhlet apparatus with anhydrous ether.

Testing the Purity of Apiol.—G. Francois (*Journal de Pharmacie and Pharm. Central-Halle*).

Atomic Weight of Tungsten.—R. Schneider.—The mean from all the author's experiments is 184.01.

Atomic Weight of Helium.—M. A. Langlet (*Zeit. Anorg. Chemie*).—The mol. of helium, like that of argon, contains only one atom, and its atomic weight must be taken as = 4.

MISCELLANEOUS.

Harben Gold Medal.—At a meeting of the Harben Nomination Committee held last month in connection with the British Institute of Public Health, Professor Max von Pettenkofer, Scientific Director of Liebig's Extract of Meat Company, was nominated Harben Gold Medallist for 1897. The medal was founded in 1895 by Henry Harben, Esq., J.P., for the recognition of eminent service to the public health, and one presentation is made every year.

Institute of Chemistry.—January Examinations, 1897—Candidates who passed the practical examination for the Associateship:—Walter Harry Barlow, Finsbury Technical College; Charles Beavis, Ph.D. (Würzburg), Universities of Bonn and Würzburg, and with Dr. Quirin Wirtz, F.I.C. (for Fellowship); Cecil Joslin Brooks, King's College, London; Ernest Mostyn Hawkins, Finsbury Technical College, London; Charles J. S. Makin, Royal College of Science, London, and the Fresenius Laboratory, Wiesbaden; William Moore, Laboratories of the Pharmaceutical Society, University College, London, and with Professor John Atfield, F.R.S., F.I.C.; Sigmund Salomon G. Rosenblum, University of Warsaw, Fresenius Laboratory, Wiesbaden, and with Dr. Samuel Rideal, F.I.C. **Intermediate Examination.**—Reginald Arthur Berry, Cambridge University; John Alfred Foster, under the late Dr. A. Norman Tate, F.I.C., Professor V. B. Lewes, F.I.C.; Robert Dexter Littlefield, University College, London, and also with Dr. John Muter, F.I.C., and Mr. W. A. H. Naylor, F.I.C. **Final Examination for the Associateship** (In Branch A., Mineral Analysis).—Frank Collingridge, B.Sc. (Lond.), University College, London (passed "Intermediate," July, 1896); William Ranson Cooper, M.A., B.Sc. (R.U.I.), Royal University of Ireland, and King's College, London; John Allsopp Walker, B.A. (Oxon.), Oxford University. (In Branch E., Analysis of Water, Food, and Drugs).—Raymond St. George Ross, Owens College, Manchester, and Dresden Polytechnic (for Fellowship). **Examiners:** Professor Percy F. Frankland, F.R.S., F.I.C., and Otto Hehner, Esq., F.I.C.

City and Guilds of London Institute for the Advancement of Technical Education.—The following is the Scheme for the Leathersellers' Company's Research Fellowships in Chemistry, founded 22 December, 1896, during the Mastership of Dr. W. H. PERKIN, F.R.S. :—

I.—Number and Amount of Fellowships.

1. The Grant of £150 a year offered by the Leathersellers' Company shall be applied in founding one or more Fellowships, entitled "Leathersellers' Company's Research Fellowships," for the encouragement of Higher Research in Chemistry in its relation to Manufactures.

2. The amount of the grant attached to each Fellowship shall be determined by the Executive Committee of the Institute, regard being had as far as practicable to the nature of the Research, the time required to complete it, and the merits of the candidate; subject in all things to the approval of the Company.

II.—Conditions of Eligibility.

1. Applications for Fellowships shall be made in writing, addressed to the Honorary Secretary of the Institute, at the Head Office, Gresham College, E.C., and shall state the name of the proposed research and the qualifications of the candidate.

2. The Fellowships shall be open to natural born British subjects, who are—

a. Students of the Institute who have completed a full three years' course of instruction in the Chemical Department of the Central Technical College, or

b. Candidates duly qualified in the methods of Chemical Research in its relation to manufactures, without restriction as to age or place of previous study, but preferably to class (a).

III.—Award.

1. The Fellowships shall be awarded by the Executive Committee with the consent of the Company in accordance with this Scheme, or with such modifications as the Company may from time to time approve.

2. The Executive Committee shall appoint a special Committee to receive applications, and select candidates, and to report thereon, and upon the progress of researches; and such Committee shall include the representative or representatives for the time being of the Company on the Executive Committee.

3. The Executive Committee shall report to the Company the award of each Fellowship, and at the close of each session shall report the results or progress of the Research or Researches undertaken during the session.

IV.—Tenure.

1. Every Fellowship shall be tenable for part of a year or for one year, and may be renewed for a second or third year, but in no case shall be held for a further period.

2. Holders of Fellowships shall devote their whole time to the prosecution of research, unless otherwise sanctioned by the Executive Committee, and shall report as required on their work.

3. The Researches shall be carried out at the Central Technical College, and the holders of Fellowships shall be subject to the regulations of the College and the supervision of the Board of Studies.

4. The Company reserve to themselves the right at any time to modify this Scheme, or to withdraw all or any of the Fellowships.

F. A. ABEL, Chairman of the Executive Committee.
JOHN WATNEY, Honorary Secretary.

MEETINGS FOR THE WEEK.

- MONDAY, 8th.—Society of Arts, 8. (Cantor Lectures). "Material and Design in Pottery," by Wm. Burton, F.C.S.
- TUESDAY, 9th.—Royal Institution, 3. "Animal Electricity," by Prof. A. D. Waller, F.R.S.
- Society of Arts, 8. "Lithography as a Mode of Artistic Expression," by George McCulloch.
- WEDNESDAY, 10th.—Society of Arts, 8. "The Chemistry of Tea," by David Crole.
- THURSDAY, 11th.—Royal Institution, 3. "The Problems of Arctic Geology," by J. W. Gregory, D.Sc., F.R.S.
- Society of Arts, 4.30 (at Imperial Institute). "The Progress of Science Teaching in India," by Prof. J. C. Bose.
- Society of Arts, 8. "The Mechanical Production of Cold," by Prof. James A. Ewing, M.A., F.R.S.
- FRIDAY, 12th.—Royal Institution, 9. "Recent Advances in Seismology," by Professor John Milne, F.R.S., F.G.S.
- SATURDAY, 13th.—Royal Institution, 9. "The Growth of the Mediterranean Route to the East," by Walter Frewen Lord.

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PATENTS, DESIGNS, AND TRADE MARKS ACTS, 1883 TO 1888.**NOTICE IS HEREBY GIVEN, that**

HENRY ROBERT ANGEL, of 7, St. Helen's Place, London, E.C., has applied for leave to amend the Specification filed in pursuance of the Application for Letters Patent, No. 335, of 1896, for "Improvements in the Manufacture of Caustic Soda, Carbonate of Soda, and Sulphide of Sodium."

Particulars of the proposed amendments were set forth in the Illustrated Official Journal (Patents), issued on the 27th January, 1897.

Any person, or persons, may give notice of opposition to the amendment (on Form G) at the Patent Office, 25, Southampton Buildings, London, W.C., within one calendar month from the date of the said Journal.

(Signed) H. READER LACK,
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THE CHEMICAL NEWS.

VOL. LXXV., No. 1942.

NOTE ON THE LIMIT OF ACCURACY ATTAINABLE IN COLORIMETRY.

By CHARLES W. FOLKARD.

As the result of a large number of experiments in 1893 and 1896, the author has come to the conclusion that analytical processes depending on the exact imitation of the colour of a solution of unknown strength by another solution containing a known amount of the substance (as in Nesslerising, &c.), are capable of affording results of far greater accuracy than has been generally supposed.

By taking proper precautions it is possible to work colorimetrically to within one-fortieth or one-fiftieth (say, 2 per cent), and by averages to within 1 per cent of the quantity of substance present (e.g., copper in ammoniacal solution).

This was at first so startling that publication was deferred, but further experience has afforded ample confirmation as regards azure blue, yellow, and red solutions, and the object of this note is to ascertain if other observers (more particularly works' chemists) have been led to the same conclusion in the course of their daily practice.

Baling, February 5, 1897.

THE EFFICIENCY OF THE HERMITE BLEACHING SOLUTION.

By CLAYTON BEADLE.

As far as I am aware there are no published results of experiments upon the bleaching efficiency of the Hermite bleaching solution in comparison with that of a solution of bleaching-powder upon the bleaching of cotton and linen fibre. All the published results—viz., those of Messrs. Cross and Bevan, and Professor Pictet—are, I believe, upon the bleaching of wood pulp.

I made a series of experiments to determine whether the Hermite solution still gave the same efficiency when used for bleaching cotton and linen rags and rag half-stuff. For this purpose a small beater was used, similar in construction to the paper-maker's hollander. I took half-stuff produced from second quality linen rags and from second quality cotton rags. Before doing experiments in the small beater, I mixed a known weight of each of the half-stuffs with a carefully ascertained volume of Hermite solution, also with bleaching-powder solution, the chlorine strength of each having been carefully ascertained.

The time required to bleach the materials to a full white was noted, and when the bleaching was complete the available chlorine in the residual liquors was determined, and the amount of chlorine consumed by the fibre calculated. The following are the results:—

Half-stuff.	PUT IN.		Strength per litre.	Per cent on fibre.
	Dry weight. Grms.	Liquor.		
1. Linen ..	162.6	Hermite ..	2.8	17.2
2. „ ..	162.6	Bleaching pd.	3.16	19.4
3. Cotton ..	176.2	Hermite ..	2.8	15.9
4. „ ..	176.2	Bleaching pd.	3.16	18.0

	Chlorine. Grms.	CONSUMED.		Time of bleaching.
		Chlorine. P.c. on fibre.		
1.	2.44	1.5		30 mins.
2.	3.72	2.29		4 hrs.
3.	4.0	2.27		2 hrs.
4.	6.49	3.68		10 hrs.

The efficiency of chlorine in the Hermite liquid as compared with that of chlorine in ordinary bleaching-powder is claimed by the inventors to be as 5 is to 3. This has been substantiated by the results of Messrs. Cross and Bevan, and Professor Pictet.

3 : 5 :: 1 : 1.66.

Comparing this with the experiments above,—

	Hermite.	Bleaching powder.			
1 and 2.	1.5	2.35	::	1	1.54
3 and 4.	2.27	3.68	::	1	1.65

These results therefore confirm fairly closely those of other observers. The next two experiments were done with a view of finding how long the Hermite solution took to exhaust itself if the chlorine put in was the exact amount necessary, according to the above experiments, to do the bleaching. The rate of bleaching was much slower than if the chlorine had been used greatly in excess. After three days, however, the liquid only contained the least possible trace of chlorine, and the fibre appeared to be perfectly bleached.

The preceding experiments were all done with "still" liquor. In the following experiments the half-stuff was put into the small beater, and the Hermite liquor allowed to flow round the beater, and washed out again by means of a washing drum, from whence it was delivered to a store tank and then again to the beater. The total amount of liquor was first of all measured both into the beaker and into the store tank, from which a sample was taken and tested for chlorine. This experiment was not done under the most favourable circumstances, as the liquor was drawn from the store tank and not from the electrolysing tank whilst the electrolysis was going on, which I think would have made a considerable difference to the results.

Half-stuff.	PUT IN.		Strength per litre.	Per cent on fibre.
	Weight dry fibre. Grms.	Volume of liquid. Grms.		
Linen ..	542	56.12	2.64	27.3
Cotton ..	470	56.12	2.64	31.5

Weight of chlorine.	CONSUMED.		Time.
	Per cent on fibre.		
5.6	1.03	40 mins.	
5.6	1.20	60 „	

It is evident that the circulating liquor is more economical than the non-circulating.

Half-stuff.	Non-circulating chlorine consumed.	Circulating chlorine consumed.	Saving by circulating over non-circulating.
Linen ..	1.5	1.03	30 p.c.
Cotton ..	2.27	1.20	47 „

My results confirm those of other observers as regards the rapidity of bleaching by the Hermite solution, which I found to bleach very rapidly, doing as much work in thirty minutes as bleaching-powder solution of the same strength would do in three hours. I also found that Hermite solution will bleach in one treatment, when any amount of bleaching-powder will fail to do so without an intermediate acid treatment. It can be used either circulating or stored in tanks for use like ordinary bleaching-

powder, but the latter, as we have seen, does not give such good results.

Laboratory, West Street, Erith,
January 23, 1897.

VISCOSE AND VISCROID.*

By CLAYTON BEADLE.

IN August, 1894 (*Journal of the Franklin Institute*, cxxxviii., No. 824), I had the honour of reading a paper before the Franklin Institute upon some new cellulose derivatives which had been discovered and partly worked out by my colleagues, Messrs. C. F. Cross and E. J. Bevan, and myself. Mr. Arthur D. Little, of Boston, took up the subject on this side of the water, and the samples which we showed you at that time were produced by him. Since then we have added considerably to our knowledge of these derivatives. The basis of these products is a substance to which we have given the name "Viscose." Viscose is chemically cellulose xanthate, the preparation and constitution of which is fully explained and set forth in my previous paper (*vide ut supra*).

The dry regenerated cellulose obtained from the viscose solution we have named "Viscoid."

It is impossible, in a paper of this length, to give a history of all the work we have done in the last two years in connection with viscose; so I have chosen to confine myself to certain branches of the work, the description of which, I trust, will prove interesting to the members of the Institute.

Manufacture of Alkali Cellulose.

In the manufacture of viscose on a large scale, we first endeavoured to discover what materials could be utilised, and in the course of our work we found that any kind of cellulose could be used, provided that it was fairly pure. The fibres, however, should be very short indeed; the process depends as much upon the length of the fibre as upon the purity of the cellulose. As an instance of this, if raw cotton be used without any disintegration it is almost impossible to mercerise it and convert it into viscose; but if the fibre be disintegrated so as to break up the ultimate fibre into pieces of about one-twentieth of the length of the original, the mercerisation and conversion into viscose is rapid and complete. With regard to the purity of the fibre, bleached wood generally yields a better viscose than unbleached wood, but it is next to impossible to convert mechanical wood (i. e., wood disintegrated by mechanical means, and containing a large amount of resin and other impurities) into viscose. The alkali cellulose often requires to be kept for several days before treatment with carbon bisulphide; but when the disintegration of the fibre is thorough, and the mixture with caustic properly effected, the maturing of the alkali cellulose is almost unnecessary.

One great precaution, which at first we lost sight of, was to prepare the alkali cellulose without contact with the atmosphere.

This we discovered by analysing a number of samples. We found that on an average about 50 per cent of the alkali had been carbonated during mercerisation, and thus rendered useless for the reaction. Alkali cellulose is converted by the action of the CO_2 of the atmosphere into sodium carbonate and cellulose without our knowing it. This change was the cause of a large number of failures, which necessitated conducting a series of trials under varying conditions, in which we determined the amount of alkali carbonated.

At last we arrived at a method of producing the alkali cellulose by which only 5 per cent to 10 per cent of the total soda is converted into carbonate.

Yield of Viscoid.

A great deal of work has been done upon the amount of viscoid yielded by different celluloses. Pure cotton yields somewhat more than its own weight. This is due to a change in the cellulose molecule (*vide Beadle, Journ. of the Franklin Inst.*, cxxxviii., No. 824). Wood, on the other hand, even when thoroughly bleached and pure, undergoes a considerable loss, which amounts often to 20 per cent. This is due to the formation of soluble products during mercerisation.

We have followed this very carefully, and it appears that bleached wood pulp contains oxycellulose, which is largely dissolved by caustic soda. Under certain conditions the regenerated cellulose has no strength, as when films made from viscose solution are found to be rotten. Very great care has to be taken in the manufacture of viscose to insure that the regenerated cellulose is not injured by the treatment. A knowledge of this can be acquired only by experience, and those who are thoroughly acquainted with the manufacture of viscose can often tell at a glance, from the appearance of the alkali cellulose or the viscose solution, whether a satisfactory product will be obtained. These conditions are now well understood by us, so that we can insure that the cellulose is always regenerated in the proper physical condition required for the particular purpose to which it is to be applied.

Production of Veneers.

Mention was made in my previous paper (*Journal of the Franklin Institute*, vol. cxxxviii., No. 824) of cutting pieces from the coagulum and annealing them under pressure for the production of sheets. This has been worked at on a larger scale. The viscose has been run into rectangular moulds, and the coagulation of the mass has been effected by exposure to a hot damp atmosphere. These masses, weighing from 250 to 400 pounds, have been dehydrated by exposing them to an atmosphere which is gradually raised in temperature. When the blocks have been sufficiently hardened, they are cut into sheets about 24 x 18 inches, by a guillotine specially constructed for the purpose. By an automatic gear the bed on which the guillotine rested was made to travel forward at every stroke of the knife, and the amount of travel could be regulated at will, so that sheets of any thickness could be cut. By this means we were able to cut about twenty sheets per minute.

The sheets were deprived of the chemical by-products, and then submitted to heavy pressure, by means of which the cellulose hydrate was dehydrated down to a compact sheet of cellulose. We had a difficulty by this method in obtaining our dehydrated sheets free from structure. They had a tendency to split in laminæ. The fracture had every appearance of slaty cleavage (*CHEMICAL NEWS*, vol. lxx., p. 139), and the planes of cleavage were found always to be at right angles to the direction in which the pressure was applied. With thin sheets we had less trouble, and I believe the reason of this was that the sheet was thinner than the laminæ. In order to avoid this difficulty with thicker sheets, we were obliged to dehydrate the coagulum to a greater extent, by exposure to a hot atmosphere of steam before applying the pressure. The fact of pressure on the coagulum giving rise to slaty cleavage, prevented us from applying pressure for moulding solid articles from the coagulum. This is obvious when we take into consideration the fact that the dehydrated laminæ formed on the outer surfaces by the first application of pressure, prevents the egress of moisture from the interior, and, as it were, seals the interior from further dehydration. We found it next to impossible to mould articles of any thickness under pressure from the coagulum for this reason, even on the application of a pressure of several tons to the square inch. When, however, the sheets are almost dried by exposure to warm air, or by one of the processes described subsequently for the production of solids, they can be embossed and

* *Journal of the Franklin Institute*, January, 1897.

stamped into small articles, such as buttons, which are sufficiently structureless for all practical purposes.

When the sheets are completely dried they offer too great a resistance for moulding under pressure. Viscoid sheets can be produced by the above process in almost any colour, either translucent or opaque, and they can be prepared in such a way that they are either soft and pliable, or stiff and horny like celluloid. Under pressure they can be embossed in various patterns.

(To be continued).

THIRD ANNUAL REPORT OF COMMITTEE ON ATOMIC WEIGHTS.

RESULTS PUBLISHED DURING 1895.*

By F. W. CLARKE.

TO THE MEMBERS OF THE AMERICAN CHEMICAL SOCIETY.

YOUR committee upon atomic weights respectfully submits the following report, summarising the work done in this branch of chemistry during 1895—a year which may be well called eventful in the history of the science. Two new elements, argon and helium, have been made known to the world, and from the most unexpected sources; the collective works of Stas have been published by the Belgian Academy, as a monument to his memory; Prof. Morley's great research upon oxygen is at last finished; and a goodly number of other important determinations have appeared. Incidentally, but pertinently, I may also call attention to the Marignac memorial lecture by Clève (*Journ. Chem. Soc.*, June, 1895), in which the atomic weight researches of the former chemist are well outlined; and to the extraordinary number of papers upon the periodic law, which have been called out by the discovery of argon and helium. These papers fall outside the scope of this report, and they are numerous enough to almost warrant a bibliography of their own.

The H : O Ratio.—Prof. Morley's work upon this fundamental constant has been published in full by the Smithsonian Institute,† and divides itself naturally into four parts:—First, the density of oxygen; second, that of hydrogen; third, the volumetric composition of water; and fourth, its gravimetric synthesis.

For the density of oxygen, or rather the weight of 1 litre at 0°, 760 m.m., at sea-level, and in latitude 45°, three sets of measurements are given, with the following mean values in grms. :—

Series 1		1.42879	± 0.000034
„ 2		1.42887	± 0.000048
„ 3		1.42917	± 0.000048

As the third series, on experimental grounds, is regarded by Morley as the best, he assigns it double weight, and on this basis the general mean of all three becomes—

$$1.42900 \pm 0.000034.$$

For the weight of a litre of hydrogen under similar standard conditions, five series of determinations are given, as follows:—

Series 1		0.089938	
„ 2		0.089970	
„ 3		0.089886	± 0.0000049
„ 4		0.089880	± 0.0000088
„ 5		0.089866	± 0.0000034

The hydrogen of the first and second series was probably contaminated by traces of mercurial vapour, and

these results are therefore rejected by Morley. For the third, fourth, and fifth series the electrolytic gas was occluded in palladium and transferred to the measuring globes without the intervention of stopcocks; thus avoiding contact with mercury and leakages of external air. Their general mean is—

$$0.089873 \pm 0.0000027.$$

Dividing the weight found for oxygen by this value for hydrogen the ratio becomes—

$$15.9002.$$

For the volumetric ratio O : 2H, Morley finds the value 1 : 2.00269. Applying this as a correction to the density ratio, we have for the atomic weight of oxygen—

$$O = 15.879.$$

In his synthesis of water Morley differs from all of his predecessors in that he weighed both constituents separately, and also the water formed. In other words, his syntheses are complete, and take nothing for granted. The weights in grms. are as follows:—

	O used.	H used.	Water found.
1	 25.9176	3.2645	29.1788
2	 25.8531	3.2559	29.1052
3	 30.3210	3.8193	34.1389
4	 30.5294	3.8450	Lost
5	 30.4700	3.8382	34.3151
6	 30.5818	3.8523	34.4327
7	 30.4013	3.8297	34.2284
8	 30.3966	3.8286	34.2261
9	 30.3497	3.8225	34.1742
10	 30.3479	3.8220	34.1743
11	 29.8865	3.7637	33.6540
12	 30.3429	3.8211	34.1559

From these data two sets of values for the atomic weight of oxygen are derivable; one from the ratio H : O, the other from the ratio H : H₂O. These sets are subjoined.

	H : O.	H : H ₂ O.
1	 15.878	15.877
2	 15.881	15.878
3	 15.878	15.873
4	 15.880	—
5	 15.877	15.881
6	 15.877	15.876
7	 15.877	15.875
8	 15.878	15.879
9	 15.879	15.881
10	 15.881	15.883
11	 15.881	15.883
12	 15.882	15.878
Mean..	.. 15.8792	15.8785

From the density work the value found was 15.879, and the mean of this with the two synthetic results is—

$$O = 15.8789.$$

Hence, for all practical purposes, the atomic weight of oxygen may be put at 15.88, with an uncertainty of less than one unit in the second decimal.

It is impracticable, in a report of this kind, to go into the details of so elaborate an investigation as this of Morley's, and a bare statement of results must suffice. The research, however, is one of the most perfect of its kind, every source of error having been considered and guarded against, and it will doubtless take its place in chemical literature as a classic. Independently of its main purpose, the book is almost a manual on the art of weighing and measuring gases, and no experimenter who engages upon work of that kind can afford to overlook it.

More recently still, a new determination of the atomic weight of oxygen has been published by Julius Thomsen (*Ztschr. Anorg. Chem.*, xi, 14), whose method is quite novel. First, aluminum, in weighed quantities, was dis-

* Read at the Cleveland Meeting, December 31, 1895. From the *Journal of the American Chemical Society*, xviii., No. 3.

† "On the Density of Oxygen and Hydrogen, and on the Ratio of their Atomic Weights." By Edward W. Morley. Smithsonian Contributions to Knowledge, 1895. 4to. xi. + 117 pages, 40 cuts. Abstract in *Am. Chem. Journ.*, xvii., 267 (gravimetric); and *Ztschr. Phys. Chem.*, xvii., 87 (gaseous densities); also note in *Am. Chem. Journ.*, xvii., 396.

solved in caustic potash solution. In one set of experiments the apparatus was so constructed that the hydrogen evolved was dried and then expelled. The loss of weight of the apparatus gave the weight of the hydrogen so liberated. In the second set of experiments the hydrogen passed into a combustion chamber in which it was burned with oxygen, the water being retained. The increase in weight of this apparatus gave the weight of oxygen so taken up. The two series, reduced to the standard of a unit weight of aluminum, gave the ratio between oxygen and hydrogen.

The results of the two series, reduced to a vacuum and stated as ratios, are as follows:—

First, $\frac{\text{Weight of H}}{\text{Weight of Al}}$	Second, $\frac{\text{Weight of O}}{\text{Weight of Al}}$
First.	Second.
0'11180	0'88788
0'11175	0'88799
0'11194	0'88774
0'11205	0'88779
0'11189	0'88785
0'11200	0'88789
0'11194	0'88798
0'11175	0'88787
0'11190	0'88773
0'11182	0'88798
0'11204	0'88785
0'11202	
0'11204	0'88787 $\pm$ 0'000018
0'11179	
0'11178	
0'11202	
0'11188	
0'11186	
0'11185	
0'11190	
0'11187	
0'11190 $\pm$ 0'000015	

Dividing the mean of the second column by the mean of the first, we have for the equivalent of oxygen:—

$$\frac{0'88787 \pm 0'000018}{0'11190 \pm 0'000015} = 7'9345 \pm 0'0011.$$

Hence,—

$$O = 15'8690 \pm 0'0022.$$

The details of the investigation are somewhat complicated, and involve various corrections which need not be considered here. The result as stated includes all corrections and is evidently good. The ratios, however, cannot be reversed and used for measuring the atomic weight of aluminum, because the metal employed was not absolutely pure.

The Stas Memorial.—As a monument to the memory of the late Jean Servais Stas, more appropriate than statue or column of stone, the Belgian Academy has published his collected works in three superb quarto volumes.* All of his great investigations are here gathered together, and in the third volume, entitled "Oeuvres Posthumes," some hitherto unpublished data are given for the important ratio between potassium chloride and silver. These data are represented by two series: one made with a uniform sample of silver, and chloride from various sources; the other with constant chloride, but with silver of diverse origin; the aim being to establish experimentally the fixed character of each substance. The first series is complete; of the second series only one experiment was found recorded among Stas's papers.

The quantity of potassium chloride equivalent to 100 parts of silver was found to be as follows:—

69'1227
69'1236
69'1234
69'1244
69'1235
69'1228
69'1222
69'1211
69'1219
69'1249
69'1238
69'1225
69'1211

Mean of first series 69'1229
Second series . . 69'1240

These results give an effective confirmation to Stas's determinations of 1882.

(To be continued).

METAL SEPARATIONS BY MEANS OF HYDROCHLORIC ACID GAS.*

By J. BIRD MOYER.

(Concluded from p. 65).

XV.—Separation of Arsenic from Zinc.

In some preliminary work zinc oxide was treated with acid gas at 200°. It completely changed to chloride, and was not volatile. Pure zinc sulphate was used to precipitate the arsenate; it was washed, dried, and ignited to 150°. The same difficulty appeared as was encountered under iron. Zinc arsenate melts down to a liquid mass as soon as the acid gas strikes it, which is extremely hard to evaporate without spattering. A small glass cover was placed over the boat, which tended to lessen the spattering, but did not entirely prevent it.

The zinc was estimated by taking the chloride up in a little hydrochloric acid and running it down with pure mercuric oxide. It was then ignited and weighed as zinc oxide. One good result was obtained, but generally the residues of zinc contained arsenic and the results were far from being concordant.

XVI.—The Separation of Arsenic from Cobalt and Nickel.

Cobalt and nickel were precipitated as arsenates in the usual manner, with a solution of pyroarsenate.

Cobalt nitrate, a Merck preparation, was carefully purified; considerable manganese was found and eliminated.

This gave the pink salt $\text{Co}_3\text{As}_2\text{O}_8 + 8\text{H}_2\text{O}$, which was ignited to the blue anhydrous compound.

Cobalt arsenate is very readily attacked by the acid gas in the cold, yielding a pink chloride. A slight heat, not much above 120°, changed it to the blue chloride and drove out the arsenic. At first it was quickly weighed as chloride, then it was taken up in a little hydrochloric acid and evaporated down with mercuric oxide. On ignition, black Co_3O_4 was obtained and weighed.

The arsenic was eliminated as usual.

	Experiment I.	Experiment II.
	Grm.	Grm.
$\text{Co}_3\text{As}_2\text{O}_8$ taken	0'1509	0'2029
CoCl_2 obtained	0'1309	—
CoCl_2 required	0'1293	—
Co_3O_4 obtained	0'0738	0'0969
Co_3O_4 required	0'0731	0'0983
Difference	+0'0007	—0'0014
As_2O_5 obtained	0'0770	—
As_2O_5 required	0'0764	—
Difference	+0'0006	—

* From author's thesis presented to the Faculty of the University of Pennsylvania for the degree of Ph.D., 1896. From the *Journ. Amer. Chem. Soc.*, xviii., December, 1896.

* "Jean Servais Stas. Oeuvres Complètes." Edited by W. Spring. Bruxelles, 1894.

On testing the cobalt residue by the Marsh test, no trace of arsenic was found. No cobalt was found in the sublimate. Some of the first experiments gave cobalt too low; it was thought that they had been heated too high, but testing showed no volatilised cobalt.

A temperature of 125° is sufficient to drive out all of the arsenic, and at this temperature there is no danger of volatilising the cobalt.

In working with nickel, the green arsenate was simply dried in the first experiment. It therefore had the composition $\text{Ni}_3\text{As}_2\text{O}_8 + 8\text{H}_2\text{O}$.

Hydrochloric acid gas attacked it in the cold. A slight heat drives out the arsenic and moisture and leaves a salmon-coloured chloride. The nickel chloride was changed to oxide by evaporating it with nitric acid and igniting.

	Experiment I.
	Grm.
$\text{Ni}_3\text{As}_2\text{O}_8 + 8\text{H}_2\text{O}$ taken	0.1502
NiO obtained.. ..	0.0554
NiO required.. ..	0.0561
Difference	-0.0007

In Experiments II. and III. the salt was made anhydrous by ignition.

	Experiment II.	Experiment III.
	Grm.	Grm.
$\text{Ni}_3\text{As}_2\text{O}_8$ taken	0.1166	0.1040
NiO obtained.. ..	0.0577	0.0523
NiO required	0.0575	0.0513
Difference	+0.0002	+0.0010
As_2O_5 obtained	—	0.0515
As_2O_5 required	—	0.0526
Difference	—	-0.0011

The Marsh test showed no arsenic with the nickel.

XVII.—Behaviour of Minerals in Hydrochloric Acid Gas.

Niccolite.—One half gram. of the mineral was finely powdered and subjected to the action of acid gas for a day, at a temperature of 200°C . It was only very slightly affected.

A second portion was dissolved in nitric acid and evaporated down in a porcelain dish. It was then transferred to a boat and evaporated to dryness. To remove all the acid it was heated in an oven to 110° for half an hour. The dry substance was acted upon by the acid gas in the cold for five hours. It changed completely to chloride. A temperature of 150° for an hour removed all the moisture and arsenic.

The nickel chloride was evaporated down with nitric acid, ignited, and weighed as NiO. The arsenic was estimated as usual.

	Per cent.
Nickel found	43.79
Nickel calculated	43.60
Difference	0.19
Arsenic found	56.66
Arsenic calculated	56.40
Difference	0.26

Undoubtedly there is still a wide field open in regard to the behaviour of hydrochloric acid gas upon mineral species. Smith and Hibbs (*loc. cit.*) showed that mimitite lost its arsenic quantitatively, when heated in a stream of acid gas. In this laboratory others are being investigated with favourable indications. The direct employment of hydrochloric acid gas upon a powdered mineral would simplify many a tedious gravimetric process, leaving the separated elements in a desirable condition for further treatment.

In the case of a mineral such as niccolite, where it must first be decomposed with nitric acid and then transferred to a boat, the advantage is not so great. This, however, can be modified, so that the time factor is reduced and the advantage of the method still retained. Instead of using a boat, which has no advantage unless the non-

volatile chlorides are to be weighed directly, a hard glass bulb can be substituted. The mineral is placed in the bulb, dissolved in nitric acid, and evaporated down by the aid of a current of air drawn through the bulb.

The residual oxides are then separated in a stream of hydrochloric acid gas as usual.

THE DETERMINATION OF ATOMIC MASSES OF SILVER, MERCURY, AND CADMIUM, BY THE ELECTROLYTIC METHOD.*

By WILLETT LEPLEY HARDIN.

(Continued from p. 63).

PART II. (continued).

THIRD SERIES.

Experiments on Mercuric Cyanide.

A SERIES of observations was made on several organic salts of mercury with a view of selecting a compound suitable for atomic mass determinations. Mercuric acetate and other similar salts were found to be unstable in the air and unsuited for accurate analyses. Mercuric cyanide, on the other hand, was found to be perfectly stable and to form well-defined crystals.

Preparation of Hydrocyanic Acid.

Five hundred grms. of potassium ferrocyanide were placed in a two litre retort connected with a condenser. A cooled mixture of 300 grms. of pure sulphuric acid and 700 c.c. of distilled water was then poured into the retort, and the mixture carefully heated until the hydrocyanic acid was distilled over into the receiver. The product obtained was re-distilled and used immediately in the preparation of mercuric cyanide.

Preparation of Mercuric Cyanide.

Fifty grms. of mercuric oxide, prepared as already described in the experiments on mercuric oxide, were dissolved in pure warm hydrocyanic acid. The solution was then filtered and evaporated to crystallisation. The transparent crystals of mercuric cyanide which separated were dissolved in pure water and re-crystallised. The product obtained by the second crystallisation was quickly rinsed with cold water and dried for six hours in an air bath at a temperature of 50° . The crystals were then ground to a finely divided powder in an agate mortar and re-dried for twenty-four hours in an air bath at a temperature of 55° . The dry white powder was then placed in a weighing tube and kept in a desiccator.

Mode of Procedure.

The mode of procedure with mercuric cyanide was somewhat different from that of the preceding experiments, in that no potassium cyanide was used in preparing the solution for electrolysis. A weighed portion of the material was dissolved in pure water in a platinum dish. When the crystals had completely dissolved, the dish was filled to within a quarter of an inch of the top with water, after which one drop of pure sulphuric acid was added. The solution was then electrolysed, and the resulting metal weighed. The strength of the current and the time of action were the same as for mercuric chloride. In the last four experiments, where rather large quantities of mercury were deposited, the strong current was allowed to act from two to six hours longer.

The results of ten experiments on mercuric cyanide, reduced to a vacuum standard on the basis of—

* Contribution from the John Harrison Laboratory of Chemistry No. 13. From the author's thesis presented to the Faculty of the University of Pennsylvania for the degree of Ph.D.—From the *Journal of the American Chemical Society*, xviii., p. 990.

4.0	= density of mercuric cyanide,
13.59	= " metallic mercury,
21.4	= " platinum dish,
8.5	= " weights,

and computed for the formula $\text{Hg}(\text{CN})_2$, assuming 12.01 and 14.04 to be the atomic masses of carbon and nitrogen, respectively, are as follows:—

	Weight of $\text{Hg}(\text{CN})_2$, Gms.	Weight of Hg, Gm.	Atomic mass of mercury.
1	0.55776	0.44252	200.063
2	0.63290	0.50215	200.092
3	0.70652	0.56053	200.038
4	0.80241	0.63663	200.075
5	0.65706	0.52130	200.057
6	0.81678	0.64805	200.103
7	1.07628	0.85392	200.077
8	1.22615	0.97282	200.071
9	1.66225	1.31880	200.057
10	2.11170	1.67541	200.077
Mean ..	..	=	200.071
Maximum ..	..	=	200.103
Minimum ..	..	=	200.038
Difference ..	..	=	0.065
Probable error	..	=	0.005

From the total quantity of material used and metal obtained the atomic mass of mercury is 200.070.

FOURTH SERIES.

According to Faraday's law the quantities of different metals deposited from their solutions by the same current are proportional to their equivalent weights. In this series of experiments an attempt was made to determine the ratio of the atomic mass of mercury to that of silver by passing the same current through the solutions of the two metals and weighing the two resulting deposits. If the proper conditions could be obtained, this would certainly be the simplest and most direct method for comparing the equivalent weights of different metals. But so many difficulties were met that the method on the whole was not satisfactory.

In the "Revision of the Atomic Weight of Gold" (*Amer. Chem. Journ.*, xii., 182), Mallet made use of this method, and in a series of careful preliminary experiments determined the conditions most favourable to its application. From a number of experiments made by passing the same current through two different solutions of copper sulphate, using pure electrolyte copper for both anode and cathode in each solution, Mallet found:—

First.—Other conditions being the same, the difference in the quantities of metal deposited from solutions of unequal concentrations was very slight and somewhat variable, but the tendency was toward a slightly larger quantity from the more concentrated solution.

Second.—With equal quantities of metal in the two solutions, and unequal quantities of free acid, the difference in the results obtained were almost insignificant and somewhat variable in direction, the tendency being toward a slightly larger quantity from the less acid solution.

Third.—Other conditions being the same, a difference in the temperature of the two solutions invariably caused a slightly larger deposit from the cooler solution.

Fourth.—Other conditions being the same, a difference in the size of the copper plates, and hence a difference in the "current density," caused a slightly greater deposit on the smaller plate.

Fifth.—A difference in the distance between the two plates did not produce a constant difference of result, but the tendency was toward a slightly larger deposit on the cathode plate farther separated from its anode.

From the foregoing experiments it is evident that the conditions most favourable to this method are, that the two solutions should be equally concentrated, of the same

temperature, and should contain equal amounts of free acid, or when the double cyanides are used, equal quantities of free potassium cyanide. And, moreover, that the two cathodes and also the two anodes should be of the same size, and that the distance between the anode and cathode should be the same in both solutions. These conditions were closely observed throughout this work.

Arrangement of Apparatus.

The deposits in this series of experiments were made in two platinum dishes of equal capacity and equal internal area. The anode in each case consisted of a coil of rather large platinum wire, the two coils being of the same shape and size. The dishes were insulated from each other by means of two glass stands. The platinum coils were completely immersed in the solutions, and the portion of the wire near the surface of the liquid was covered with paraffin to prevent surface contact. The current, after passing through the two solutions, was allowed to pass through a hydrogen voltameter in order that its strength might be observed at any time.

In the second arrangement of apparatus the platinum dishes were made the anodes, and two pieces of platinum foil of the same shape and size were used for the cathodes. The results, however, from this second arrangement were not as satisfactory as from the first.

Mode of Procedure.

A solution of the double cyanide of silver and potassium was placed in one of the platinum dishes, and a solution of the double cyanide of mercury and potassium in the other. The quantities of silver and mercury present in their solutions were approximately proportional to their equivalent weights. Each solution contained a slight excess of potassium cyanide. The dishes were placed in their positions, and the anodes immersed some time before the current was allowed to act. When the temperature of the two solutions was the same as that of the room the connection was made, and the same current allowed to pass through the two solutions. The quantity of metal deposited was never allowed to exceed one-half of the metal present in the solution at first. Before interrupting the current, the solutions were syphoned from the two platinum dishes at the same time with two syphons of the same bore. The deposits were then washed several times with boiling water, carefully dried, and their weights determined. Experiments were made with currents of different strength, and with solutions of various degrees of concentration. The results obtained were far from being satisfactory. The strength of current which seemed best adapted to the work was that which deposited about one-tenth of a gm. of silver per hour.

From a large number of experiments, only seven results were obtained which seem of any value in determining the atomic mass of mercury. And it must be added that many others were rejected, not because they were known to be vitiated in any way, but because the results obtained for the atomic mass of mercury differed from those obtained from other methods. It is possible that, in a large number of experiments the condition would be more favourable in some than in others, but whether the close agreement of the results selected was due to this or to the balancing of errors could not be determined.

Seven results computed on the basis of 107.92 for the atomic mass of silver are as follows. (See next column).

Computing from the total quantities of mercury and silver obtained we have 199.971 for the atomic mass of mercury.

Although the cause of the large variation in the rejected observations could not be definitely determined, several sources of error suggest themselves.

First, small quantities of hydrogen were undoubtedly set free in the process of electrolysis, and unless these quantities were always equal in the two solutions, which is not probable, an error would be introduced.

Second, in some solutions an error might easily be

	Weight of Hg. Grm.	Weight of Ag. Grm.	Atomic mass of mercury.
1	0.06126	0.06610	200.036
2	0.06190	0.06680	200.007
3	0.07814	0.08432	200.021
4	0.10361	0.11181	200.011
5	0.15201	0.16402	200.061
6	0.26806	0.28940	199.924
7	0.82808	0.89388	199.929
Mean ..	..	..	= 199.996
Maximum ..	..	..	= 200.061
Minimum ..	..	..	= 199.924
Difference ..	..	..	= 0.137

introduced by a change in the atomicity of mercury, but in a solution of the double cyanide of mercury and potassium this change is hardly probable.

Third, the occlusion of hydrogen by the two metallic deposits would also be a possible source of error; but only small errors could be introduced in this way.

To account for the difference of several units in the results, the source of error first mentioned seems by far the most probable.

Summary.

In the discussion of the results obtained in the different series of observations on the compounds of silver, the probable sources of error and likewise the advantages of the method were pointed out. The same discussion applies equally well to the observations on mercury.

It is evident that the first three series of observations on mercury are entitled to more weight than the last series. Just why the results on mercuric bromide should be lower than those on mercuric chloride is not clear. Both compounds are certainly well adapted to atomic mass determinations, inasmuch as they can be purified by both crystallisation and sublimation. The most probable impurity in mercuric bromine would be mercuric chloride, but that would tend to increase rather than lower the results. The series of observations on mercuric cyanide have, perhaps, one advantage over the others, in that no potassium cyanide was used. The results obtained in this series are still higher than those obtained from mercuric chloride, and almost two-tenths of a unit higher than those obtained from mercuric bromide. However, as the same care was exercised in the purification of the material for each of the three series, and as there was no apparent error in either case, equal weight must be given to each of the three series in determining the most probable value of the atomic mass of mercury. And, as the mean of the last series is almost identical with the mean of the first three, equal weight can be given to this series without introducing any error.

Computing the general mean from the separate observations we have:—

	Atomic mass of mercury.
First series	200.006
Second „	199.883
Third „	200.071
Fourth „	199.996

General mean = 199.989

From the total quantities of material used and metal obtained, the general mean is:—

	Atomic mass of mercury.
First series	199.996
Second „	199.885
Third „	200.070
Fourth „	199.971

General mean = 199.981

Combining this with the first general mean we have:—

	Atomic mass of mercury.
First general mean	= 199.989
Second „ „	= 199.911

Most probable mean of all the results = 199.985
or 200 for the atomic mass of mercury.

(To be continued).

ALUMINUM ANALYSIS.*

By JAMES OTIS HANDY.

(Concluded from p. 68).

Hydrated Alumina.

HYDRATED alumina is analysed for water, silica, and sodium carbonate.

Water.—Ignite 1 grm. in a closely covered crucible, at first gently and then intensely for fifteen minutes over the strongest blast. The loss on ignition includes water and the carbon dioxide of the sodium carbonate. Calculate the carbon dioxide from the sodium oxide found and deduct it from the loss on ignition.

Silica.—Hydrated alumina is soluble in sulphuric acid of 42° B. The silica, however, is left undissolved. 42° B. sulphuric acid is made by mixing 900 c.c. of concentrated sulphuric acid with 1290 c.c. of water. Five grms. of hydrated alumina are treated with twenty-five c.c. of 42° B. sulphuric acid and heated until the alumina appears to be all dissolved. Dilute to 100 c.c. and boil. Filter, wash, ignite, and fuse the residue with one grm. of potassium bisulphate and cool. Dissolve in water, filter, wash, ignite, and weigh in crucible, treat with sulphuric acid and hydrofluoric acid, evaporate, ignite, and weigh again. Loss equals silica.

Soda.—The method of the determination of soda is the same in calcined and hydrated alumina. The method is that of J. L. Smith, and is described under "Sodium in Aluminum." Calculate sodium chloride to sodium carbonate, if the sample is hydrated, and to sodium oxide if the sample is calcined alumina.

Calcined Alumina.

Water and soda are determined as in hydrated alumina.

Silica.—Fuse 1 grm. of the finely ground alumina with 10 grms. of potassium bisulphate. If this does not make a clear fusion add 2 grms. of bisulphate and heat up again. Dissolve the fusion when cool in water and filter. Burn off the insoluble residue. Fuse it with 1 grm. of sodium carbonate and cool in fifteen c.c. of water in a four-and-a-half-inch evaporating dish. Add twenty-five c.c. of 25 per cent sulphuric acid. When all soluble matter has dissolved, remove the crucible and evaporate down until sulphuric acid fumes escape. Cool, dilute with water, boil, filter, ignite, and weigh silica plus crucible, treat with sulphuric and hydrofluoric acids, and weigh again. Loss equals silica.

Analysis of Bauxite.

(Method adopted, May, 1895.)

No unusual apparatus or reagents are required.

One and five-tenths grms. of very finely ground bauxite (previously dried at 100° C. and bottled), is taken for analysis. Weigh into a five-inch porcelain evaporating dish and dissolve in fifty c.c. of acid mixture. This mixture is the same as that used for aluminum analysis. Boil the solution down until fumes escape and keep the residue fuming strongly for about fifteen minutes. Cool, add 100 c.c. of water, stir and then boil for ten minutes. Filter, wash well with water, receiving the filtrate in a beaker of about 300 c.c. capacity. The filtrate and washings

* From the *Journal of the American Chemical Society*, Sept., 1896.

should amount to about 175 c.c.: Burn off the insoluble residue (which consists chiefly of silica, with a little titanic acid, oxide of iron, and alumina) and weigh it in the crucible, add three drops of 25 per cent sulphuric acid and about five c.c. of hydrofluoric acid and evaporate slowly to dryness. Ignite very strongly and weigh. The loss in weight equals silica. Add to the residue in the crucible 1 gm. of potassium bisulphate and fuse quickly and thoroughly over a Bunsen burner, cool and place the crucible in the beaker containing the main sulphuric acid solution. The small residue from this fusion will be silica, and is to be added to the silica already found. Having obtained the sulphate solution containing all the alumina, ferric oxide, and titanic oxide, make it up to 550 c.c. and mix. Then fifty c.c. will equal three-tenths gm. bauxite. Take fifty c.c. and dilute to 300 c.c. Add two c.c. of concentrated hydrochloric acid and ammonia in slight excess, boil for five minutes, let the precipitate settle, filter, and wash very thoroughly with hot water. Test the filtrate for further alumina by boiling. Burn off the filter paper and ignite the precipitate very strongly after crushing all the lumps of alumina. Weigh alumina, ferric oxide, and titanic oxide.

Titanic Acid.—Take 100 c.c. of the original sulphate solution (six-tenths gm.), add ammonia until a slight permanent precipitate is formed, then add sulphuric acid from a pipette or burette until this precipitate just re-dissolves. Finally add 1 c.c. more of 25 per cent. sulphuric acid and dilute to 400 c.c. If the bauxite is high in iron (which will be indicated by the distinct yellow colour of this solution) sulphur dioxide gas must be run into it until it is decolourised and smells strongly of sulphur dioxide, but if the solution is nearly colourless, indicating a low percentage of iron, only sulphur dioxide water need be used for the reduction. Boil well for one hour, adding water saturated with sulphur dioxide occasionally. Filter off the titanic oxide through double filters, and wash well with hot water. If the precipitate is yellow, indicating the presence of iron, it can be fused with 1 gm. of potassium bisulphate, the fusion dissolved in water, and the iron determined in this solution by reducing with zinc and titrating with permanganate. This is not often necessary.

Oxide of Iron.—Take 50 c.c. of the sulphate solution, add 10 c.c. of dilute sulphuric acid, and 1 gm. of granulated zinc, and set the beaker in a warm place. When reduced, filter and titrate the iron with standard potassium permanganate. More zinc is used for bauxites high in iron.

Method for Iron Determination, using a larger Quantity of Bauxite. (Applicable to Purest Ores).

Place a half gm. of the finely powdered ore in a large platinum crucible, and add 3 c.c. of 25 per cent. sulphuric acid and 5 c.c. of hydrochloric acid, and evaporate very slowly to fumes; drive off the excess of sulphuric acid by heat, boil out the residue with water, and add 10 c.c. of dilute sulphuric acid. Remove the crucible and reduce with zinc, as above, and titrate.

Water and Organic Matter.—Ignite three-tenths gm. cautiously at first and finally very strong in a covered crucible. The loss of weight equals water and organic matter.

ON THE ACTION OF WAGNER'S REAGENT UPON CAFFEINE, AND A NEW METHOD FOR THE ESTIMATION OF CAFFEINE.*

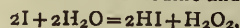
By M. GOMBERG.

THE use of iodine in potassium iodide as a general qualitative reagent for alkaloids dates as far back as 1839

(Bouchardat, *Comp. Rend.*, ix., 475). It was, however, R. Wagner (*Dingl. Poly. Journ.*, clxi., 40; *Ztschr. Anal. Chem.*, i., 102) who first employed it for the quantitative estimation of vegetable bases, and this solution has since been known as Wagner's reagent. He based his conclusion upon trials with solution of quinine and cinchonine, showing that under approximately similar conditions they always require the same amount of iodine for complete precipitation. Hence empirical factors could be established which would enable one to use a standard solution of iodine for the titration of all such alkaloids as form insoluble superiodides. The method, however, was not frequently employed, for the reason that there was no experimental proof as to the constancy of composition of the precipitates. Moreover, it was noticed that some of the precipitates give up a portion of their iodine to water, i.e., they are not completely insoluble. Hence concordant results could not be obtained. Later, Schweissinger (*Arch. d. Pharm.*, lxiv., 615, 1885) applied this method to the estimation of strychnine and brucine. His results have led him to the conclusion that while the method is very satisfactory for strychnine, it is far from being so for brucine. Recently Kippenberger (*Ztschr. Anal. Chem.*, xxxiv., 317; xxxv., 10), in his research upon the isolation and separation of alkaloids for toxicological purposes, has reviewed the subject of the action of Wagner's reagent upon alkaloids, and gives considerable prominence to this as one of the best methods for the estimation of the vegetable bases. His method of procedure was practically the same as that first proposed by Wagner. The alkaloid is dissolved in acidulated water, and to the solution a tenth or twentieth normal solution of iodine in potassium iodide is gradually added until all the alkaloid is precipitated and the supernatant liquid shows a slight excess of iodine. Instead of filtering and washing the precipitate, as was done by Wagner and Schweissinger, Kippenberger allows the precipitate to settle, and either decants or filters off an aliquot portion of the mother-liquid for the estimation of iodine not taken up by the alkaloid. The estimation of iodine is always done by means of a standard solution of sodium thiosulphate.

It has been usually assumed, for reasons not entirely clear, that the composition of the precipitates is Alk.HI.I_2 , i.e., diiodides of the hydriodides of the alkaloids are formed. Of the three atoms of iodine only two can be estimated directly by titration with sodium thiosulphate. The hydriodic acid is supposed to come from the potassium iodide, while the two "superiodine" atoms are furnished by the free iodine dissolved in the potassium iodide. The quantity of an alkaloid precipitated by a known volume of Wagner's reagent is calculated on this assumption, 2I: molecular weight of alkaloid :: amount of iodine taken up: x = amount of alkaloid. Schweissinger found that the method of calculation agrees entirely with the theoretical figures for strychnine. Kippenberger has called into question the correctness of this mode of calculation. He, too, assumes that the composition of the precipitates is to be represented by the formula Alk.HI.I_2 , but he claims that all three atoms of iodine are supplied by the free iodine, and none by the potassium iodide. Therefore the calculation of the amount of alkaloid precipitated is to be done, according to Kippenberger, by the use of the proportion, 3I: molecular weight of alkaloid :: amount of iodine taken up: x = amount of alkaloid.

The hydriodic acid, it is supposed by Kippenberger, results from the interaction of iodine and water,



a reaction which is facilitated or induced by the avidity of the alkaloids to form insoluble periodides of the hydriodides. His reasons for assuming that such a peculiar reaction takes place under the simple conditions of precipitation are too lengthy to be given here. All his arguments rest upon the assumption that all alkaloids form

* From the *Journal of the American Chemical Society*, xviii., No. 4.

periodides of uniform composition, Alk.HI.I_2 , and that the same alkaloid gives always the same periodide. Now, there is no reason, *a priori*, why this should be the case. Jörgenson's (*Journ. Prakt. Chem.*, 1870-78, [2], ii., 433, &c.) extended researches show that different alkaloids, when treated under apparently the same conditions, give periodides of entirely different compositions. Thus, morphine gives with Wagner's reagent Alk.HI.I_3 (Jörgenson, 1870, *Journ. Prakt. Chem.*, [2], ii., 438); codeine furnishes with excess of Wagner's reagent Alk.HI.I_4 ; and caffeine, as will be shown, gives Alk.HI.I_4 , &c. It is safe to say that not until we ascertain exactly the composition of the different periodides as produced under the conditions of titrations, will the use of Wagner's reagent for quantitative purposes be placed upon a sound basis.

I have dwelt at such length upon this subject, because the method for the estimation of caffeine presently to be described, is based upon experimental evidence which is entirely contradictory to Kippenberger's conclusions. Whatever the cause may be with other alkaloids, his theory as to the production of hydriodic acid from iodine and water does not hold good in the case of caffeine.

Wagner, in describing his method, gives a list of alkaloids which are completely precipitated by iodine solution, and also mentions that "caffeine, theobromine, piperine, and urea are not precipitated at all" (*loc. cit.*, 41). His statement, so far at least as caffeine is concerned, has stood since then uncontradicted. It has found its way not only into standard treatises and text-books,* but even into periodical literature of recent date. As late as 1894, Kunze (*Ztschr. Anal. Chem.*, xxxiii., 23), in reviewing the chemistry of caffeine and theobromine, calls attention to this peculiarity of the two alkaloids. The non-precipitation of caffeine by Wagner's reagent has come to be recognised as a distinguishing feature of this alkaloid from almost all other vegetable bases.

And yet this is entirely contrary to actual facts. Instead of forming an exception, caffeine conforms to all the requirements† necessary in the application of this test. It is well known that most of the alkaloids as such are insoluble, or only very slightly soluble in water; they require the presence of some acid for their complete solution. In other words, alkaloids in the form of their salts are soluble in water. Whenever Wagner's reagent is applied for the precipitation of an alkaloid, it is always applied to a solution of some salt of it, preferably acidulated with sulphuric or hydrochloric acid. Therefore, even when strictly neutral salts of alkaloids are employed, there is still the possibility of the formation of hydriodic acid, or rather of the hydriodides of the alkaloid, as, for instance, $\text{Alk.HCl} + \text{KI} = \text{Alk.HI} + \text{KCl}$. The hydriodide thus produced is at once precipitated as a periodide. Now, it so happens that caffeine is tolerably soluble in water, and it has become customary to work with solutions of caffeine as a free alkaloid, and not in the form of its salts. The question as to whether solutions of free alkaloids are precipitated with Wagner's reagent has not, to my knowledge, been studied. My preliminary experiments in that direction show that at least some alkaloids (morphine, atropine, strychnine, &c.), are precipitated. I have not examined yet whether these periodides are identical in composition with those produced from the salts of the alkaloids. But so far as caffeine is concerned, it is true that a neutral solution of it gives no precipitate when treated with a solution of iodine in potassium iodide. When, however, the addition of Wagner's reagent is either followed or preceded by the addition of some dilute acid, there is at once thrown down a dark-reddish pre-

cipitate, remaining amorphous even on long standing.* The composition of this periodide is, as will be shown, $\text{C}_8\text{H}_{10}\text{N}_4\text{O}_2.\text{HI.I}_4$. It was obtained for analysis in many different ways—by using either caffeine or iodine in excess, and by employing different acids. The periodide produced is, however, always of the same composition. The precipitates were allowed to settle, filtered by means of a pump, washed with water to remove the excess of potassium iodide, dried on porous plates, and finally in a vacuum over sulphuric acid.

(To be continued).

SOME ANALYTICAL METHODS INVOLVING THE USE OF HYDROGEN DIOXIDE.†

By B. B. ROSS.

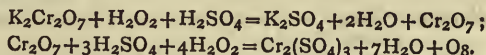
THE use of hydrogen peroxide as a laboratory reagent, although originally restricted to a few operations of minor importance, has within recent years met with a much wider extension, and its numerous applications in both qualitative and quantitative analysis render it at present almost indispensable in every well-equipped analytical laboratory.

Among the more interesting applications of this substance in quantitative estimations are those which are based on the reaction which takes place when an excess of hydrogen dioxide is brought in contact with an acid solution of chromic acid, and Baumann (*Zeitschr. Anal. Chem.*, xxxi., 436) several years since described quite fully a number of analytical processes growing out of the reaction referred to.

In the process for the estimation of chromic acid in soluble chromates as outlined by Baumann, the substance under examination is first brought into a state of solution, and the not too concentrated liquid is transferred to a generating flask of special construction.

Ten c.c. of dilute sulphuric acid are next added, after which from 5 to 10 c.c. of commercial hydrogen peroxide are run in from a small closed vessel connected with the generating flask, while the oxygen which is evolved, after the vigorous shaking of the contents of the flask, is collected over water in an azotometer.

The following equations given by Baumann illustrate the chemical changes connected with the above-described reaction:—



From these equations it will be seen that for 2 molecules of chromic acid, or 1 molecule of potassium dichromate, there are evolved 8 atoms of oxygen, giving an equivalent of 445.3 c.c. of oxygen (measured at 0° C. and 760 m.m. pressure) for each grm. of chromic acid which may be present.

The writer, soon after the appearance of the original article by Baumann, made a number of experimental tests of this method, with a view to applying it to some other analytical processes, and still more recently has conducted a series of tests for the purpose of determining the adaptability of Baumann's method to the indirect volumetric estimation of iron.

In the dichromate method for the volumetric determination of iron, as commonly employed, the end-point

* Prescott, "Organic Analysis," p. 80; Allen, "Comm. Organic Analysis," iii., (2), 481; Flückiger, "Reactions" (Nagelvoort's Translation), p. 26; not affected by Wagner's reagent in either neutral or acid solutions; Dragendorff, 1888. Ermittlung von Giften says that caffeine gives a dirty-brown precipitate. From the text it is not improbable he used iodine in hydriodic acid.

† This test is perhaps most frequently made in a neutral solution, representing, as customary state free caffeine and normal salts of other alkaloids.

* Almost the same can be said of theobromine, making allowance for the difference of solubility of the alkaloid in water. A saturated solution of it (containing one part of theobromine to 1600 of water) gives no precipitate with Wagner's reagent, but on the addition of a drop of acid there separates in a short time a crystalline periodide. Contrary to usual statements, I find that theobromine in acid solutions gives a heavy precipitate with Wagner's reagent, of a peculiar dirty-blue colour.

† Read at the Buffalo Meeting, August 22, 1896. From the *Journal of the American Chemical Society*, xviii., p. 918.

of the oxidation process is ascertained by the reaction with potassium ferricyanide.

As the end of this reaction is almost invariably difficult to determine, particularly if zinc has been employed as a reducing agent, the dichromate process has met with but limited application.

In order to apply the principle of the chromic acid method of Baumann to the estimation of iron, an excess of dichromate solution was employed in all of the tests and experimental determinations, the amount of the excess of chromic acid being determined by the volume of oxygen evolved upon treatment with hydrogen dioxide.

The mode of procedure adopted was as follows:—

A dichromate solution was prepared by dissolving 4.913 grms. of C. P. crystallised potassium dichromate in water and diluting to a bulk of 1 litre.

The iron solution employed in standardising the dichromate and permanganate solutions was obtained by dissolving iron wire in dilute sulphuric acid, the solution being reduced with metallic zinc, as usual, previous to titration.

The dichromate solution was also titrated against a freshly-prepared solution of ammonium ferrous sulphate, the strength of which had been determined by titration with permanganate solution, which had also been carefully standardised by means of iron wire.

In order to ascertain the strength of the dichromate solution by the hydrogen dioxide method, about 15 c.c. of the dichromate solution is run into the generating flask above referred to, and there is also added an amount of ferric sulphate solution (free from ferrous sulphate) equivalent to about 0.06 to 0.10 grm. of iron. The object of employing the ferric sulphate in this standardisation is to supply approximately the same conditions as obtain in the process for the actual determination of iron.

The amount of oxygen given off from chromic acid in the presence of ferric sulphate is slightly less than that evolved when ferric sulphate is absent, but the amount of ferric iron present may vary considerably without affecting the volume of oxygen liberated.

To the contents of the generating vessel about 10 c.c. of dilute sulphuric acid are now added, and the flask is then connected by means of a rubber tube with a Schulze's azotometer, which has been filled with water to the zero point.

From 5 to 10 c.c. of hydrogen dioxide are next run in from a small closed vessel connected with the generating flask, and the mixed liquid is then shaken, at first gently, and afterwards vigorously. The tube leading from the flask to the azotometer should be provided with a stop-cock, which should be closed before and opened immediately after each shaking.

The last trace of the oxygen liberated will not be disengaged until after the lapse of about five minutes, but it is not necessary to continue the shaking during the whole of this period. After equalising the height of the water in the two tubes of the azotometer, the volume of oxygen is noted, and is easily corrected for temperature and pressure by reference to proper tables.

In order to test the strength of the dichromate solution by means of iron wire, a given weight of the wire is dissolved in dilute sulphuric acid, the solution reduced with zinc, as usual, and rapidly transferred to the generating flask (filtering, if necessary).

An excess of dichromate solution is now run in, hydrogen dioxide is added, and the oxygen is set free and collected as before described.

If a large excess of dichromate has been used in the preliminary test, duplicate tests should be made with employment of a small excess, say from 2 to 3 c.c. of the dichromate.

The strength of the solution can then be readily calculated by difference, and, if necessary, the results can be checked by still further tests.

In the determination of iron in ores by this process, the solutions of ferric iron are reduced by zinc, as in the

common permanganate method, and the remainder of the process is conducted just as described for the standardisation of the dichromate by means of iron wire.

In addition to numerous tests of solutions of pure iron, several estimations of iron in iron ores were made by this process, the results obtained being compared with those secured by the permanganate method.

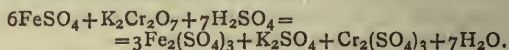
The following are the results of the tests of the iron ores referred to:—

	Permanganate method. Mean of several determinations.	Dichromate method.
Iron ore No. 1 ..	40.92	40.59 41.25
Iron ore No. 2 ..	54.71	55.35 55.43 55.50

In the determination of iron in ores by this process, it is best, as in the case of the tests with iron wire, to employ only a small excess of the dichromate solution, after making a preliminary determination, as the results are much more accurate with a small than with a large excess of chromic acid.

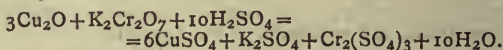
While a sufficient number of determinations have not been made to ascertain the probable value of this method as an independent process for the estimation of iron, nevertheless some of the results secured would seem to warrant the conclusion that it might prove of utility as a check method, it being easy of execution and not at all time-consuming.

The following equation represents the changes which take place when the dichromate is brought in contact with the iron solution after reduction:—



The writer has also attempted to apply the principle of the chromic acid method above described to the estimation of invert sugar, or rather to the determination of the amount of cuprous oxide thrown down from Fehling's solution in the process commonly employed for estimating reducing sugars.

The following equation represents the changes which take place when cuprous oxide is brought in contact with potassium dichromate in the presence of dilute sulphuric acid:—



The cuprous acid thrown down from the sugar solution under examination is brought upon an asbestos filter connected with a filter-pump, and thoroughly and rapidly washed with hot water. The filter and contents are next transferred to the generating flask of the apparatus before described, and after the addition of dilute sulphuric acid an excess of dichromate is run in.

Very thorough and long-continued agitation of the contents of the flask is necessary in order to effect the complete oxidation and solution of the cuprous oxide, and the hydrogen peroxide must not be added until the solution is complete.

The oxygen liberated on the addition of the hydrogen dioxide is collected, and the volume noted as before described. The equivalent amounts of chromic acid, cuprous oxide, and invert sugar can be easily calculated from the data thus secured.

This method, while apparently satisfactory from a theoretical standpoint, has so far failed to give sufficiently uniform results, one of the chief objections to the process being the difficulty attendant upon the solution of the cuprous oxide.

With improvements in the details of manipulation of the process, however, it is quite possible that more satisfactory results could be obtained.

NOTICES OF BOOKS.

The University Tutorial Series. The Tutorial Chemistry. Part I.—Non-metals. By G. H. BAILY, D.Sc., Ph.D. Heidelberg, Lecturer on Chemistry in the Victoria University. Edited by WILLIAM BRIGGS, M.A., F.C.S., F.R.A.S., Principal of University Correspondence College. London: W. B. Clive, University Correspondence College Press. Warehouse, 13, Booksellers Row, Strand, W.C. Pp. 226.

We must confess our partial inability to explain the nature or characteristics of "tutorial chemistry." We know that certain Universities, such as, e.g., Oxford and Cambridge, call themselves "tutorial," whilst others, such as Munich and Heidelberg, rank as investigational, and one at least, that of London, is purely examinational. But how any given Science, or modification of a Science, can be called tutorial we doubt. From our inspection of the volume before us we should think that tutorial chemistry must approximate closely to examinational chemistry, or to the preparatory work required for passing examinations. If the Correspondence College facilitates the process of "passing," and thus helps to shake public faith in the Chinese system of higher education now dominant in Britain, it will have done a valuable and needful work.

The author and editor very justly contend that it is unwise in the earlier stages to overburden the student with chemical theory. They are also, in our opinion, right in referring the study of the principles of light, heat, and electricity to works on physics.

The leading truths and laws of chemistry are here expounded in a most masterly manner; made, in fact, accessible to very moderate capacities, if only perseveringly and honestly applied to the task.

There is no attempt to introduce novel matter or to initiate the student into research, either of which aim would have been distinctly outside the plan of the work, if not outside the entire scope, of the University Correspondence College. Within that scope, if we rightly apprehend it, a better manual could scarcely be written.

The Australian Medical Directory and Handbook; including a Short Account of the Climatic and Sea-side Health Resorts in Australia, Tasmania, and New Zealand. Edited and Compiled by LUDWIG BRUCK, Fourth Edition, corrected up to September, 1896. (Copyright). Sydney: L. Bruck, Medical Publisher. London: Baillière, Tindall, and Cox. 1896.

THE scope of this useful work is somewhat wider than it would appear from the title. It comprehends also Fiji and the British portion of New Guinea. We are glad to find that the author calmly ignores all the non-qualified practitioners, who are now so numerous.

There is an abstract of the Medical Acts of Australia, Tasmania, New Zealand, and Fiji. There is a list of the medical and allied scientific societies in Australasia. Some of them are of high standing, and have done good work. Such are, e.g., the Royal and the Linnean Societies of New South Wales, the former of which bodies has a medical and a microscopical section. The Intercolonial Medical Congress of Australasia is an itinerant body after the pattern of the British Association.

The scale of fees legally recoverable by medical men are certainly not exorbitant, and, in comparison with the wages of mechanics, servants, &c., may be called paltry. In the local Medical Directory for each Colony, the altitudes to the various towns above sea level are given, and a rough view of the racial character of the population. We are sorry to see to what an extent the "heathen Chinese" predominates in some localities. An interesting feature is the chapter on health-resorts. Here we find given the average rainfall, the extreme and

average temperatures, and the kind of constitution for which they are most adapted.

At some of the littoral towns sea-bathing is mentioned as one of the local attractions or beneficent features. The abundance or scarcity of sharks in the sea—a point of great importance—is not mentioned. At some places a range for bathing is fenced in with iron chains.

City and Guilds of London Institute for the Advancement of Technical Education. Examinations Department. Report of the Work of the Department for the Session 1895-96. Exhibition Road, London, S.W. 1896.

THE activity of the Examination Department is greater and wider than we might expect from its unhappy name. It includes the arrangement of courses of instruction in technical subjects, the consideration of the qualifications of teachers.

A distinction seems to have been made between the certificate granted to students who have attended a course of lessons at a recognised school and that given to candidates who produce no evidence of such training. The results are said to have been very satisfactory.

The total number of subjects is 62, but in five of these no examinations have actually been held. Of the greatest percentage of failures, viz., 76, 1 per cent was in lithography, and the smallest, 0.00, were in the alkali and soap manufacture, in the lace manufacture, and in ship carpentry.

At Aylesbury there was one candidate for examination, who passed.

La Unifikazion da las Medidas. Par K. NEWMAN. Valparaiso: Karlos Kabazon. 1897.

CHILI is a perfect focus of reforms, especially orthographic. Senhor K. Newman's publications remind us of our old friend the *Phonetic Nuz*. The present object of the author is to advocate the metric system "pure and simple." He urges its adoption by all nations in its original polysyllabic nomenclature, in preference to the more convenient form devised in Holland.

The names proposed by the French revolutionary commission may be unobjectionable for wholesale transactions, but for the retail business of daily life they have proved themselves impracticable.

We should recommend K. Newman, when advocating any reform, to do so in a language "understood of the people," and not bring it in a novel and fantastic orthography.

MISCELLANEOUS.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on Feb. 1st, Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, presiding. The following were elected Members:—Mr. Alfred Louis Cohen, Mrs. Delaforce, Sir Charles A. Elliott, K.C.S.I., LL.D., Mr. John Lawson Johnson, Dr. A. Liebmann, Mr. T. George Longstaff, Mr. Howard Marsh, F.R.C.S., the Rev. E. G. C. Parr, M.A., Mr. Charles Rose, and Mr. Edward P. Thompson. The special thanks of the Members were returned to Sir Frederick Abel, Bart., K.C.B., for a donation of £50, and to Mr. J. Wolfe Barry, C.B., for a donation of £25 to the fund for the promotion of Experimental Research at Low Temperatures.

NOTES AND QUERIES.

* * Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Epsom Salts.—A correspondent asks whether Epsom salts are best reduced to the state of a dry powder by hot cylinders or in an oven, and then rolling in a mill.

MEETINGS FOR THE WEEK.

- MONDAY, 15th.—Society of Arts, 8. (Cantor Lectures). "Industrial Uses of Cellulose," by C. F. Cross, F.C.S.
Society of Chemical Industry, 8. Adjourned Discussion on Mr. W. J. Dibdin's Paper on "The Character of the London Water Supply."
- TUESDAY, 16th.—Royal Institution, 3. "Animal Electricity," by Prof. A. D. Waller, F.R.S.
Society of Arts, 8. "The Progress of Canada during the past Sixty Years of Her Majesty's Reign," by Joseph G. Colmer, C.M.G.
- WEDNESDAY, 17th.—Society of Arts, 8. "Light Railways," by Everard R. Calthrop.
- THURSDAY, 18th.—Royal Institution, 3. "The Problems of Arctic Geology," by J. W. Gregory, D.Sc., F.R.S.
Chemical, 8. "The Oxidation of Sulphurous Acid by Potassium Permanganate," by T. S. Dymond and F. Hughes. "Sodamide and some of its Substitution Derivatives," and "Rubidamide," by A. W. Titherley, M.Sc., Ph.D.
Society of Arts, 8. "The Mechanical Production of Cold," by Prof. James A. Ewing, M.A., F.R.S.
- FRIDAY, 19th.—Royal Institution, 9. "The Approaching Return of the Great Swarm of November Meteors," by G. Johnstone Stoney, M.A., F.R.S.
- SATURDAY, 20th.—Royal Institution, 3. "The Growth of the Mediterranean Route to the East," by Walter Frewen Lord.

TO CORRESPONDENTS.

- R. G. B.—We do not think any notice of the mentioned patents has yet appeared.
- C. Stanley.—The responsibility of advising in such a case is more than we care to take.
- A. S. Chase.—Consult "Explosives and Their Powers," by Col. J. P. Cundill, and "A Handbook of Modern Explosives," by M. Eissler.

CIVIL SERVICE COMMISSION.

FORTHCOMING EXAMINATION.

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at the ARTILLERY COLLEGE (20–25), 24th February.
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HOW SOON SHALL THE STUDENT BEGIN THE STUDY OF QUALITATIVE ANALYSIS?

By ALFRED C. BEEBE.

IN the following paper I shall attempt to show at what stage of laboratory work the student can most advantageously be introduced to the study of qualitative analysis.

To the teachers who begin the laboratory course with qualitative analysis I have nothing to say. But to the teachers who require the student to devote all, or a large part, of the first months in the laboratory to the study of the preparation and properties of the non-metallic elements, I wish to present some arguments for replacing that department of the subject by the study of qualitative analysis.

I have three objections to requiring the student beginning laboratory work to perform experiments relating to the non-metallic elements. First, the experiments are more or less dangerous. Second, the student nearly always fails to connect the different experiments with each other. Third, the work does not awaken and hold the student's interest as well as qualitative analysis does. Of course, the student must have a good knowledge of the properties and preparation of the non-metallic elements, but it seems to me this is best gained from lectures, illustrated by the proper experiments given by the teacher before the entire class. I wish to say that I think this part of the subject is often made so unnecessarily complex that the student's ideas of it are very confused and hazy. Let the first ideas of chemistry, both in the lecture room and the laboratory, be as simple as the subject will permit. The details are easy enough to learn afterwards if they be necessary.

Let me consider at greater length the three objections made above to the study in the laboratory of the non-metallic elements by the beginner.

First: Danger. A university professor said to me recently, "I always dread hydrogen day; I am nervous every minute the students are working with this dangerous gas, for I feel as if a serious explosion might occur at any time when students have as little experience as the beginner possesses." This state of mind is, I believe, not uncommon among teachers who require their students to experiment with hydrogen soon after beginning the laboratory work. I feel sure that almost every teacher knows of several serious explosions with hydrogen, and of many more that might have been serious except for good luck. Considering these facts it seems to me unwise to expose the student to this unnecessary danger, for no amount of chemical knowledge can repay a pupil for the loss of his eyesight. The danger is, however, much lessened by some months' experience in the laboratory, and then explosive gases may be given to the pupils should the teacher think fit to do so. The preparation of oxygen is attended with more or less danger, and I know of several serious accidents from this cause. Phosphorus is not a substance to trust a beginner with, and yet I know some widely-used text-books which give this dangerous substance to the pupil soon after his entrance to the laboratory. One text-book even goes so far as to give work with the iodide of nitrogen!

The handling of acids, the use of chlorine, bromine, &c., make trouble enough without adding work on explosive gases like hydrogen, or work with dangerous substances like phosphorus.

Qualitative analysis, on the other hand, has no experiments that are even moderately dangerous. Of course, dangerous experiments may be included in the branch of the subject, but they are unnecessary, while for the non-metallic elements they form an essential part of the course.

Second: Unconnectedness. Although the experiments on the non-metallic elements are connected it is very difficult, in my experience, to make the student see that connection. For instance, after oxygen is experimented with no further work on that subject is required, and the teacher must be continually asking review questions if the subject is to remain fresh in the student's mind. After several weeks' experimenting has been done, the amount of back work becomes too large for the teacher to review, and as the experiments do not require repetition, those on one element seem to have almost no connection with those on other elements. I have taken some trouble to look into this matter, and have heard so many students complain about it, that I consider it an important point. Qualitative analysis, on the other hand, requires that experiments once learned be remembered, for the future work uses them repeatedly, since each day's work in a good text-book on this subject is closely related to what has gone before and what is to follow. This fact and the repetition necessary while analysing unknown substances oblige the student to remember his work, and he goes out of the laboratory with a scheme for analysis containing facts as valuable as those relating to the non-metallic elements, fixed with reasonable firmness in his mind, instead of a confused mass of material such as the work on the non-metallic elements too often leaves. Even reasonable repetition will not fix seemingly unconnected facts, while connected phenomena almost fix themselves in the mind.

Third: Interest. In no part of the work in the chemical laboratory is so much interest shown as in the analysis of unknown solutions and substances. This one fact would, to me, be sufficient argument for the use of qualitative analysis for the student's first laboratory work. It were a waste of words for me to say that we should endeavour to awaken and sustain the student's interest in a subject as much as possible, for every teacher knows how smoothly and advantageously things run when a genuine interest is taken in any sort of work or study. From my experience, as student and teacher, I feel perfectly confident that qualitative analysis is much more fitted to do this than the study of the non-metallic elements. The student feels he is a real chemist as soon as he analyses his first "unknown solution." Be that solution ever so simple he realises that he has acquired a new power, and in most cases he becomes anxious to increase it. This feeling of independence is helped if no results to experiments are given in his text-book, for he thereby becomes an original investigator, and not merely a student who is required to perform certain experiments which shall agree with his text-book. This attitude sharpens the observing and reasoning faculties immensely. The interest shown in qualitative analysis is oftentimes amazing. For example, I have often seen the majority of a university class work from four to six hours *extra* a week simply from interest in this branch of the subject. I know of one teacher whose student became so interested in qualitative analysis, that in self-defence he had to refuse to remain in the laboratory more than two hours after the regular hour for closing had come. This last happened in a public high school, with a class of boys and girls averaging sixteen years old, where we surely ought to find average work. Some of this interest may be due to the teachers, but from the fact that it is not an uncommon experience, I am inclined to think that much of it is due to the fascination of qualitative analysis. I have never seen any such results from the study of the non-metallic elements.

Summing up the advantages of the study of qualitative analysis for the beginner, when compared with the study

of the non-metallic elements, I think I may say that the former is less dangerous, more connected, and holds the student's interest much better.

Savanna, Ill.

VISCOSE AND VISCOID.*

By CLAYTON BEADLE.

(Concluded from p. 75).

Production of Viscoid.

For Use in Turning, &c.—Viscoid is prepared by coagulating the viscose in the same way as described above for the production of veneers or sheets, but the moulds are circular instead of rectangular. As might well be supposed, the difficulties of coagulating the viscose increase with the diameter of the cylinder. Great care has to be taken in the early stages to prevent the material from cracking. Immediately after coagulation takes place there is a considerable shrinkage, and unless the coagulation proceeds uniformly throughout the mass there is danger of it cracking. The time of coagulation increases with the diameter. The moulds in which the material is coagulated are specially constructed, and made of a material which is unaffected by the viscose, and the interior surface has to be such that the coagulum does not adhere to it, otherwise it is sure to crack on shrinking. After setting, the solid cylinders of coagulum are removed from their moulds. At this stage they are elastic and pliable, somewhat like rubber, but have to be handled with great care. They consist of about 15 per cent of cellulose, 7 per cent of chemical by-products, and 78 per cent of water of hydration.

Washing.—They are next placed in water for the removal of the by-products. We have done much work to determine the best conditions, and the cheapest and most effective method for getting rid of the by-products.

When the cylinders of coagulum are placed vertically in tanks provided with false perforated bottoms and immersed in water, the by-products are found to diffuse out and creep down the sides of the coagulum and to accumulate in the space below the false bottom. This action is so perfect that often, when the water is undisturbed, the top portion contains only a trace of by-products and the lower portion is heavily charged with them. The removal of by-products is accelerated by the use of hot water. We find that the time required for the removal of the by-products varies as the square of the diameter of the coagulum. During washing the coagulum undergoes a further shrinkage, so that when the washing is complete the coagulum contains about 20 per cent of cellulose.

Drying.—The washed coagulum is next removed from the tanks and dehydrated or dried. The dehydration of the coagulum has presented us with some very interesting problems, which we have to a large extent solved. When the washed material is exposed to the air, it gradually contracts and loses weight, and this goes on until it is finally deprived of moisture, and nothing is left but the dry viscoid. We found it difficult to ensure that the material should retain its proper shape on drying. With "rack" or air drying at from 90° to 110° F., a solid cylinder remains flat at the ends, provided that the length is four times that of the diameter. If it is less than this it has a tendency to become concave on the ends, and it becomes more concave up to a certain point as the length diminishes in proportion to the diameter. The sides of a solid cylinder have a tendency sometimes to become convex in proportion as the ends become concave. Unless drying is uniform, a cylinder of 1½ inches diameter, and less, will bend less towards the driest side. With larger cylinders this is not so noticeable. When cylinders are

placed upon a rack that is not perforated, the top end contracts and dries the most rapidly, so that the cylinder becomes tapered towards the top. When the rack is perforated so as to allow of the free passage of air, the cylinder is sometimes found to be smallest at the bottom. Sometimes the cylinders are irregular in diameter or warped in final drying. During the process of drying the coagulum is in a semi-plastic state, and when the drying is uneven is under considerable stress. The viscoid is caused to flow in the direction to relieve the stress, so that, when finally dried and rigid, it is found to have assumed its original shape. It generally, however, leaves some marks of its temporary distortion. The time of drying, so far as our determinations have gone, varies as the square of its diameter.

When hot air is used the rate of drying cannot be increased beyond a certain degree without serious injury to the solid. The solid, by constant exposure to hot air, becomes skin-dried, whilst the interior remains comparatively moist. The outer dried skin offers a great resistance to the passage of further moisture, and so the drying is very much retarded. If, under these conditions, the moisture is able to escape from the interior, the exterior—being dry and consequently rigid—offers great resistance to the contraction of the interior, and the consequence is that fracture often takes place. If the drying be not quite so rapid as to produce fracture, the interior is under tremendous stress, and, when the cylinder is cut in section, cleavage often takes place in the direction of the length of the cylinder. When, however, the drying is conducted much slower, the moisture—which is always tending to pass from the wetter to the drier portions—finds a way of escape, and is removed from the surface by the atmosphere. We have been able to overcome these difficulties, and the product obtained is almost free from structure. The washed or unwashed coagulum has a natural inclination to contract when surrounded by a hot medium, even when the same has no drying capacity whatever. The rate of contraction increases with the temperature; thus, a coagulum containing 10 per cent of cellulose will rapidly contract to about half its bulk on immersion in boiling water. When heated in this way there is no indication of case hardening. There is a limit, however, to this contraction. We tried the effect of saturated steam at different pressures upon cylinders of washed coagulum, which contained about 15 per cent of cellulose. The pressure was varied from 3 to 8 atmospheres; the time of exposure was one hour in each case. The average contraction was about 25 per cent. The experiments go to show that there is little or no difference between the 3 atmospheres or 8 atmospheres on the amount of contraction. As the diameter increases the rate of contraction diminishes. The next set of trials were made to determine what effect time had upon the shrinkage in an atmosphere of saturated steam at 4 atmospheres. Taking the original weight as 100, after one hour's treatment, the weight became 82.9.

After two hours, the weight became	74.5
" three " " "	65.7
" four " " "	63.0.

The above was on a piece of washed coagulum, containing about 15 per cent of cellulose. We next took some unwashed coagulum, containing about 11 per cent of cellulose, and suspended it in an atmosphere of saturated steam at 4 atmospheres. It lost 44.8 per cent during the first hour, 14.7 during the second hour; therefore it had contracted during the two hours' treatment to 40.5 per cent of its original weight. A receptacle was placed immediately beneath the coagulum to catch the by-products, which were found to be recovered without dilution. With blocks of double the diameter of the above, i. e., 75 m.m., the weight after two hours' treatment was 56.6 per cent of the original. The effect of saturated steam is to produce a rapid contraction of the coagulum, if it contains from 10 to 15 per cent of cellulose,

* Journal of the Franklin Institute, January, 1897.

and this contraction is rapid until a concentration of about 20 per cent of cellulose is reached. By a prolonged treatment in saturated steam the coagulum gradually contracts until it contains about 25 per cent of cellulose, beyond which point it does not alter.

Ivory coloured Viscoid.

Ordinary viscose has about the colour and consistency of molasses, and yields a viscid which has the general appearance of horn. We set to work to produce viscid in imitation of ivory, and whilst doing this we discovered a new combination of viscose, which gave rise to a creamy white solution. This was found to last longer in the liquid form than the ordinary viscose. On drying down it yielded a product which is a very fair substitute for ivory. It has a specific gravity of 1.8 to 1.85. We had several sets of billiard balls turned from this, and they were found to play very well indeed. The angle, however, at which the balls left each other after striking was somewhat less than that of ivory balls. With the plain viscid balls, which have a sp. gr. of 1.5, the angle is greater than that of ivory, and we believe it possible to mix these two compositions in such a way that both the specific gravity and the angle at which they leave each other after striking resemble that of ivory. This "ivory" viscid has been used for various articles, such as acorns for blind-cords, knife-handles, brush-backs, &c.

Viscid for Electrical Work.—The plain viscid was carefully tested to determine its insulating properties. It is about equal to vulcanised fibre for insulating purposes. It appears that if great care is taken to thoroughly remove the last traces of by-products, and to well season the viscid, the insulating properties can be made much superior to vulcanised fibre. Cellulose, when completely deprived of moisture, is almost a perfect insulator, and its insulation diminishes as the hygroscopic moisture increases. Viscoid will probably replace vulcanised fibre to a large extent for electrical work, but at present it is very much inferior to vulcanite. It is likely that we shall find some means of improving it, so that it may be made to replace that substance.

Black Viscoid.—We next set to work to prepare an ebony-black viscid, and after about six months' work we have obtained a uniform black viscid, which in some respects appears to be superior to the plain substance. The ebony viscid will probably have a greater range of utility than any of the other products we have obtained. A jet-black is preferable to white or any colour for machine-tool handles, &c.

Blue Viscoid.—We have obtained a somewhat more expensive product, very much resembling *lapis lazuli*. This is very nice for turning and carving, and takes a very high polish, and looks very well when made into fancy articles, such as umbrella and walking-stick handles.

Various Colours.—We have now got viscid in a large variety of colours and shades, and have also succeeded in producing grained and mottled effects, which have a very pretty appearance when turned and polished. We see no reason why viscid should not be used in place of celluloid for many purposes. Some samples have been kept two or three years, and they do not discolour. Keeping the viscid for a long time appears rather to improve it than otherwise.

Paper Sizing.

Mr. Little mentioned this application of viscose in his paper (*Journal of the Franklin Institute*, cxxxviii., No. 824). Viscose is now being extensively used in paper-mills for this purpose. It does not size the paper in the sense that rosin does, although when viscose is used a large proportion of the rosin can be dispensed with. It is added to the beater, and a chemical substance is afterwards added which precipitates the cellulose among the fibres as a flocculent mass. The effect that it has upon the paper is, of course, dependent upon the amount used.

It strengthens the paper from 30 to 100 per cent, and the paper produced with viscose, besides being much stronger, is also harder, has a better "feel" and rattle, and admits of a much better surface when calendered. It also assists in the retention of clay, and prevents the loss of short fibres which, as a rule, pass through the wire cloth of the machine. It therefore gives a larger output to the machine. It is chiefly used in the manufacture of "wrappings" and bag papers, but it is coming into use also for news and other papers. Where additional strength is not required, it enables the paper-maker to use a lower-class and weaker fibrous material, without prejudicing either the strength or quality of his paper. By this means it decreases the cost of production. The precipitation of the cellulose requires a great deal of skill. Unless care is taken to ensure the right conditions, and to add the precipitating agents in the right proportions, the cellulose is precipitated as a non-adhesive mass, which gives neither strength nor hardness to the paper. It is necessary to know exactly the conditions of working in each mill before good results can be ensured, but when once these are known and understood there is no difficulty in obtaining uniform results.

The Nature of Hygroscopic Moisture in Cellulose.

The behaviour of viscid on drying in the mass opened up some very interesting problems. It appeared to throw light upon the hygroscopic moisture of celluloses. I had previously observed (*Nature*, xlix., No. 457) that the cotton fibre, when deprived of moisture (either by drying in an air-bath at 105° C., or in a desiccator over sulphuric acid) and exposed to the air, rose in temperature rapidly for about eight minutes. It reached a temperature of about 4½° F. above that of the atmosphere, where it remained nearly stationary for a few minutes. It then fell gradually, and after about seventy minutes' exposure it again reached the temperature of the surrounding atmosphere. I endeavoured to find what connection this had with the rate at which bone-dry cotton fibre assumed its hygroscopic moisture. I found that cotton fibre took about seventy minutes, and, by weighing the cotton at different intervals, and comparing the rate of gain in weight with the rate of increase in temperature, I was led to the conclusion that the two were closely connected. The same experiments were repeated with anhydrous viscid which had been ground to a powder before drying. The viscid first of all suddenly fell below the temperature of the atmosphere. It fell much slower than the cotton fibre, and even at the end of 160 minutes' exposure it was still about 3° above the atmospheric temperature. At this point it had only recovered about 90 per cent of its hygroscopic moisture, and it only came to a constant weight after about 210 minutes' exposure. By grinding viscid to a much finer powder than was used for the above, and repeating the experiments, a much more regular curve was obtained, and the hygroscopic moisture was very much diminished; but the time required for the material to fall to the temperature of the atmosphere was not lessened. I took some cotton-wool which I ground to a fine powder, and I found that, although it took about sixty minutes to recover its hygroscopic moisture, it contained only about 4 per cent instead of 7 per cent moisture. All these experiments were repeated with similar results. From this it is evident that the hygroscopic moisture of a cellulose, whether amorphous or in the fibrous condition, is dependent not only upon the character of the cellulose itself, but also upon the extent to which the cellulose is disintegrated. This is contrary to accepted views on the subject. It is only when particles of viscid are below a certain size that the hygroscopic moisture is materially diminished. It appears that the cellulose that composes the cell-walls of the ultimate fibre is under certain stress when deprived of moisture, in the same way that lumps of viscid are when they are dried, and that the amount of stress determines the amount of moisture that it will take up to

relieve the stress. Cellulose expands when hydrated, and it appears that the hygroscopic moisture is really water of hydration of the cellulose, and that it tends to hydrate in proportion to the stress that exists in the anhydrous cellulose. This accounts for the fact that the smaller particles of viscid contain less hygroscopic moisture than the larger; also, which is more marked, disintegrated cotton fibre contains much less hygroscopic moisture than when the fibre is left intact. I have prepared small particles of viscid, which, when bone-dry, would fly to pieces on being scratched, like "Prince Rupert Drops;" but the same particles, on being placed in a damp atmosphere, swell somewhat and recover their strength.

Particles can, on the other hand, be prepared in such a way that they do not exhibit brittleness, and they expand to a much less degree on being placed in a moist atmosphere. The results all tend to the same conclusion, in my mind, as to the nature of hygroscopic moisture in cellulose.

THIRD ANNUAL REPORT OF COMMITTEE ON ATOMIC WEIGHTS.

RESULTS PUBLISHED DURING 1895.*

By F. W. CLARKE.

(Continued from p. 76).

Cobalt.—The atomic weight of cobalt has been re-determined by Thiele ("Die Atomgewichtsbestimmung des Kobalts," a doctoral dissertation, Basel, 1895). First, carefully purified oxide of cobalt, CoO , was reduced in hydrogen. The weight and results are as follows:—

Residual Co.	Loss of O.	Atomic weight of Co.
0.90068	0.24429	58.843
0.79159	0.21445	58.912
1.15558	0.35716	58.788
Mean		58.848

Reduced to vacuum standards this becomes—

$\text{Co} = 58.826$,

when $\text{O} = 15.96$.

In a second method metallic cobalt was dissolved in hydrochloric acid, and the solution evaporated to dryness with special precautions against dust. The chloride thus obtained was then dried at 150° in a stream of pure gaseous hydrochloric acid, so that basic salts could not be formed. From the weight of cobalt and of cobalt chloride the ratio $\text{Co} : \text{Cl}_2$ is determined. The chlorine was afterwards re-estimated as silver chloride, giving the ratio $\text{Co} : 2\text{AgCl}$. The weights are subjoined:—

Co taken.	Cl taken up.	AgCl.
0.7010	0.8453	—
0.3138	0.3793	—
0.2949	0.3562	1.4340
0.4601	0.5657	2.2812
0.5818	0.7026	2.8303
0.5763	0.6947	—
0.5096	0.6142	2.4813

Hence, with $\text{Cl} = 35.37$, and $\text{Ag} = 107.66$, $\text{Co} =$

$\text{Co} : \text{Cl}_2$.	$\text{Co} : 2\text{AgCl}$.
58.66	—
58.52	—
58.57	58.828
58.66	58.825
58.52	58.803
58.68	—
58.69	58.750

Mean 58.64

Mean 58.801

The second column is subject to a small correction for dissolved silver chloride, which reduces the mean to $\text{Co} = 58.770$. Reduced to a vacuum this becomes 58.765, and the value from the $\text{Co} : \text{Cl}_2$ ratio becomes 58.61. Thiele regards $\text{Co} = 58.765$ as the most probable value to be derived from his experiments. This becomes—

With $\text{O} = 16$ $\text{Co} = 58.912$
,, $\text{O} = 15.88$ $\text{Co} = 58.470$

In my report for 1894 I gave Winkler's work on cobalt and nickel, which involved their ratios to iodine. In a supplementary paper Winkler (*Ztschr. Anorg. Chem.*, viii., 291) gives some similar experiments with iron, intended to show that errors due to metallic occlusion of hydrogen are absent from his determinations. He succeeds in proving that such errors, if they exist, must be very small. Thiele also considered their possibility, and guarded against them in the preparation of his cobalt.

Zinc.—Atomic weights re-determined by Richards and Rogers (*Ztschr. Anorg. Chem.*, x., 1; calculations made with $\text{O} = 16$, $\text{Ag} = 107.93$, and $\text{Br} = 79.955$), who used the bromide method. Zinc bromide, carefully purified, was treated gravimetrically with standard silver solution. The weights and results are subjoined:—

First, $\text{ZnBr}_2 : 2\text{AgBr}$.

ZnBr_2 .	AgBr .	Atomic weight of Zn.
1.69616	2.82805	65.469
1.98198	3.30450	65.470
1.70920	2.84549	65.487
2.35079	3.91941	65.470
2.66078	4.43751	65.400

Mean.. .. 65.459

Second, same ratio.

ZnBr_2 .	AgBr .	Atomic weight of Zn.
2.33882	3.90067	65.400
1.97142	3.28742	65.434
2.14985	3.58539	65.402
2.00966	3.35074	65.463

Mean.. .. 65.425

Third, $\text{ZnBr}_2 : \text{Ag}_2$.

ZnBr_2 .	Ag_2 .	Atomic weight of Zn.
2.33882	2.24063	65.409
1.97142	1.88837	65.444
2.14985	2.05971	65.396
2.00966	1.92476	65.472

Mean.. .. 65.430

Two additional series of data are given by Richards alone, as follows:—

First, $\text{ZnBr}_2 : \text{Ag}_2$.

ZnBr_2 .	Ag_2 .	Atomic weight of Zn.
6.23833	5.9766	65.403
5.26449	5.0436	65.404
9.36283	8.9702	65.392

Mean.. .. 65.402

Second, $\text{ZnBr}_2 : 2\text{AgBr}$.

ZnBr_2 .	AgBr .	Atomic weight of Zn.
2.65847	4.43358	65.410
2.30939	3.85149	65.404
5.26449	8.77992	65.404

Mean.. .. 65.406

The final mean adopted by Richards is 65.404. With $\text{O} = 15.88$ this becomes—

$\text{Zn} = 64.913$.

Cadmium.—Mr. Bucher's paper,* as its title indicates,

* "An Examination of some Methods Employed in Determining the Atomic Weight of Cadmium," by John E. Bucher. Johns Hopkins University doctoral dissertation. Baltimore (Friedenwald), 1895.

* Read at the Cleveland Meeting, December 31, 1895. From the *Journal of the American Chemical Society*, xviii., No. 3.

is a study of methods rather than a final determination of atomic weight; but the results recorded in it compare well with those reached by others. His starting-point is metallic cadmium, purified by nine distillations *in vacuo*, and from this material, with pure reagents, his various preparations were made. Vacuum weights are given, and the antecedent values used in calculation are O, 16; S, 32.059; C, 12.003; Cl, 35.45; Br, 79.95; and Ag, 107.93.

First, cadmium oxalate, dried for fifty hours at 150°, was decomposed by heat, and so reduced to oxide. The variations are mainly attributed to imperfect dehydration of the oxalate. Weights and results are as follows:—

Oxalate.	Oxide.	Atomic weight of Cd.
1'97674	1'26474	111'74
1'94912	1'24682	111'83
1'97686	1'25886	111'85
1'87099	1'19675	111'81
1'37550	0'87994	111'86
1'33313	0'85308	111'96
1'94450	1'24452	112'02
2'01846	1'29210	112'09

Mean.. .. 111'89

Second, cadmium oxalate was transformed to sulphide by heating in a stream of hydrogen sulphide. The data are:—

Oxalate.	Sulphide.	Atomic weight of Cd.
2'56319	1'84716	112'25
2'18364	1'57341	112'19
2'11643	1'52462	112'03
3'13105	2'25582	112'12

Mean.. .. 112'15

Third, cadmium chloride, dried at 300° in a stream of dry, gaseous hydrochloric acid, was precipitated by silver nitrate, and the silver chloride was collected with all necessary precautions. The weights and results are subjoined:—

CdCl ₂ .	AgCl.	Atomic weight of Cd.
3'09183	4'83856	112'34
2'26100	3'53854	112'33
1'35729	2'12431	112'32
2'05582	3'21727	112'34
1'89774	2'97041	112'31
3'50367	5'48473	112'28
2'70292	4'23087	112'30
4'24276	6'63598	112'44
3'40200	5'32314	112'37
4'60659	7'20386	112'47
2'40832	3'76715	112'42
2'19144	3'42724	112'46
2'84628	4'45477	112'32
2'56748	4'01651	112'41
2'31003	3'61370	112'41
1'25008	1'95652	112'32
1'96015	3'06541	112'47
2'29787	3'59391	112'45
1'94227	3'03811	112'42
1'10976	1'73547	112'47
1'63080	2'55016	112'48

Mean.. .. 112'39

Fourth, cadmium bromide was analysed in much the same way as the chloride. The weights and results are as follows:—

CdBr ₂ .	AgBr.	Atomic weight of Cd.
4'39941	6'07204	112'35
3'18030	4'38831	112'42
3'60336	4'97150	112'45
4'04240	5'58062	112'29
3'60505	4'97519	112'38

Mean.. .. 112'38

Fifth, cadmium sulphate was formed by synthesis from metallic cadmium. 1'15781 grms. cadmium gave 2'14776 cadmium sulphate. Hence Cd=112'35. As any impurity in the sulphate would tend to lower the atomic weight found, this is probably a minimum value.

Sixth, metallic cadmium was converted into oxide by solution in nitric acid and ignition of the nitrate. The ignition was performed in double crucibles, both porcelain in Experiments 1 and 2, the inner one of platinum in the rest of the series. Weights and results as follows:—

Cd.	CdO.	Atomic weight of Cd.
1'26142	1'44144	112'12
0'99785	1'40135	112'04
	Mean.. ..	112'08
1'11321	1'27247	111'84
1'02412	1'17054	111'91
2'80966	3'21152	111'87
	Mean.. ..	111'87

In this case additional experiments were made to discover the sources of error, leading to corrections which bring the results near to those found in the chloride and bromide series. Each of the methods is quite fully discussed, and the sources of error are noted. With O=16, 112'39 seems to be a close approximation to the true atomic weight of cadmium.

Molybdenum.—Seubert and Pollard (*Ztschr. Anorg. Chem.*, viii., 434; calculations on the basis of O=15'96), by two distinct methods, have re-determined the atomic weight of this element. First, molybdenum trioxide was dissolved, in weighed quantities, in a standard solution of caustic soda. The excess of soda was then measured by titration with standard sulphuric acid and lime water. In another set of experiments the volumetric value of the caustic soda had been estimated with standard hydrochloric acid, while the last compound had also been determined gravimetrically in terms of silver chloride. Hence the data, all considered together, give from their true end terms, the ratio MoO₃:2AgCl, although in a very indirect manner; and for this indirection the authors give good reasons. The weights and results, considering only the end terms, are as follows:—

MoO ₃ .	AgCl.	Atomic weight of Mo.
3'6002	7'1709	95'734
3'5925	7'1569	95'708
3'7311	7'4304	95'757
3'8668	7'7011	95'749
3'9361	7'8407	95'720
3'8986	7'7649	95'740
3'9630	7'8941	95'723
3'9554	7'8806	95'694
3'9147	7'7999	95'686
3'8543	7'6767	95'740
3'9367	7'8437	95'688
	Mean.. ..	95'722

Reduced to vacuum standards this becomes Mo=95'729. With O=16, Mo=95'969; and with O=15'88, Mo=95'249.

Another series of determinations, in confirmation of the first, was made by the old method of reducing molybdenum trioxide in hydrogen. The weights and results are subjoined:—

MoO ₃ .	Mo.	Atomic weight of Mo.
1'8033	1'2021	95'736
1'7345	1'1564	95'777
3'9413	2'6275	95'756
1'5241	1'0160	95'741
4'0533	2'7027	95'813

Mean.. .. 95'765

Reduced to vacuum, $Mo=95.735$, a value very close to the other. When $O=16$, the atomic weight of molybdenum is very near the even number 96.

(To be continued).

ON THE ACTION OF WAGNER'S REAGENT UPON CAFFEINE, AND A NEW METHOD FOR THE ESTIMATION OF CAFFEINE.*

By M. GOMBERG.

(Continued from p. 81).

I. This sample was obtained by slowly adding a solution of iodine in potassium iodide to a solution of caffeine acidulated with sulphuric acid. The iodine was added until the supernatant liquid was decidedly red. The whole was allowed to stand three hours, filtered, washed, and dried as described above. The total iodine was estimated in the usual way, i.e., by suspending a weighed sample in water, adding sulphurous acid solution, then silver nitrate and nitric acid; filtered, washed, and dried. The "exterior" iodine, i.e., the iodine not as hydriodic acid, was estimated by direct titration with standard sodium thiosulphate.

0.2002 grm. gave for total iodine 0.2785 grm. AgI.
0.2358 " " exterior " 0.1433 " I.

II. This sample was obtained by adding to an acidulated solution of caffeine enough iodine to precipitate about one-half of the caffeine present.

0.2563 grm. gave for total iodine 0.3591 grm. AgI.
0.1659 " " exterior " 0.0997 " I.

III. A neutral solution of caffeine was mixed with an excess of Wagner's reagent, and to the mixture dilute sulphuric acid was gradually added so long as a precipitate was produced.

0.4039 grm. gave for total iodine 0.5668 grm. AgI.
0.1424 " " exterior " 0.0866 " I.

IV. Filtrates from I. and III., on long standing, gave a deposit of dark blue needle-like crystals, which were collected, washed, and dried as before.

0.7884 grm. gave for total iodine 1.1064 grm. AgI.
0.4450 " " exterior " 0.2685 " I.

V. This was obtained by re-crystallising the amorphous precipitate from methyl alcohol.

0.2890 grm. gave for exterior iodine 0.1756 grm. I.

VI. Obtained by re-crystallising the amorphous periodide from hot ethyl acetate.

0.4807 grm. gave for total iodine 0.6709 grm. AgI.
0.2777 " " exterior " 0.1622 " I.

Calculated for	Per			Found.		
$C_8H_{10}N_4O_2.HI.I_2$	cent.	I.	II.	III.	IV.	V. VI.
Total iodine..	76.44	75.12	75.66	75.84	75.83	— 75.40
Exterior iodine	61.15	60.79	60.11	60.81	60.34	60.76 60.22

When some of the periodide is treated with a solution of sulphur dioxide, and then extracted with chloroform, it furnishes unchanged caffeine.

The composition of this periodide of caffeine appears to be different from that described by Tilden (*Journ. Chem. Soc.*, xviii., 99, 1865), which he obtained by exposing to sunlight an alcoholic solution of caffeine containing some hydriodic acid. The slow oxidation of the hydriodic acid furnished the iodine, and the compound thus obtained has the composition, according to Tilden, $2(C_8H_{10}N_4O_2.HI.I_2).3H_2O$. It is a lower periodide than the one which is obtained when iodine dissolved in potassium iodide is directly added to caffeine, as the latter has the composition $C_8H_{10}N_4O_2.HI.I_4$. Tilden also mentions that by the addition of alcoholic iodine to a solution of caffeine in weak sulphuric or hydriodic acid, he obtained a deposition of black granules, which upon analysis furnished about 75 per cent of total iodine. He says that it probably consists of a compound containing nine atoms of iodine. But there is hardly any doubt that he had the tetraiodide of caffeine hydriodide.

Properties.—When dry the periodide is a violet-blue amorphous powder melting at $213^\circ C$. When moist it rapidly loses iodine on exposure to air. It is permanent when dry, and suffers but slight loss when heated to $100^\circ C$. Two grms. heated for four hours at that temperature lost only 0.027 grm. = 1.33 per cent. It loses but very little of its iodine when suspended in water, giving up enough iodine to saturate the liquid. The presence of potassium iodide in the water favours the liberation of iodine, but even then it is but slight. The periodide dissolves readily in alcohol, especially when heated, with considerable decomposition into the free base and iodine. It is more soluble in methyl alcohol and suffers less decomposition in that solvent. It can be obtained from methyl alcohol, on spontaneous evaporation of the solvent, in the form of beautiful crystals, with a metallic dark bluish lustre. When examined under the microscope the crystals appear to consist of six-sided prisms. Ether, whether cold or warm, decomposes it but slightly. The periodide is insoluble in chloroform, carbon disulphide, and benzene. It is soluble without decomposition in hot ethyl acetate, from which it separates on cooling as a dark granular crystalline deposit, which melts at $215^\circ C$.

Limits of Precipitation.—Like most alkaloids, caffeine is precipitated by Wagner's reagent even from very dilute solutions of the base. Although not characteristic, it is yet as delicate a test for caffeine as we have. The limits of precipitation, under the influence of different acids, will appear from the accompanying table. The tests apply to 1 c.c. of the solution mentioned, acidulated with two or three drops of the acid, and to this two drops of Wagner's reagent (twentieth normal) was added.

(To be continued).

* From the *Journal of the American Chemical Society*, xviii., No. 4.

Dilution.	Sulphuric acid. 5 per cent.	Hydrochloric acid. 5 per cent.	Nitric acid. 5 per cent.	Acetic acid. 5 and 50 per cent.	Oxalic acid. 5 per cent.	Tartaric acid. 10 per cent.	Citric acid. 10 per cent.
1 : 250	Very heavy.	Very heavy.	Very heavy.	None.	Heavy.	Slight.	Faint.
1 : 1000	Very heavy.	Very heavy.	Very heavy.	—	Heavy.	Very slight.	None.
1 : 1500	Heavy.	Heavy.	Heavy.	—	Slight.	None.	—
1 : 3000	Fair.	Fair.	Fair.	—	Faint.	—	—
1 : 5000	Very slight.	Slight.	Slight.	—	—	—	—
1 : 8000	Very slight.	Slight.	Slight.	—	—	—	—
1 : 10000	None.	Very slight.	Very slight.	—	—	—	—

THE ACTION OF BORON ON IRON AND STEEL, AND ERRORS IN IRON ANALYSIS ACCOUNTED FOR BY THE PRESENCE OF THAT ELEMENT.

By H. N. WARREN, Principal, Liverpool Research Laboratory.

SOME five or six years ago a somewhat lengthy description was published in the *CHEMICAL NEWS* with respect to the action of boron upon metallic iron, which researches at that time led to the discovery of the now well-known compound boronisen.

Quite recently some rather remarkable notes have appeared in scientific literature of the preparation of that compound by the aid of electrical furnaces; it is therefore the author's intention, after a long and varied experience with the element, to deny most emphatically that anything like such a temperature as the electric arc is required in order to form a boro-ferric compound. Even ordinary ferric borate, obtained by precipitating ferric chloride by means of a soluble borate, may, after drying, be readily reduced, at a red heat, to an amorphous—and at the same time pyrophoric—boride; or may be obtained of a silvery whiteness, fusible at a white heat and containing from 6 per cent boron, by melting the above-mentioned compound under a layer of borax, the compound being of sufficient hardness to cut glass and even scratch flint, the boron being set free in the elementary condition by means of electro dissolution. This, however, is by no means the only way of impregnating iron with boron; on the other hand, it is extremely difficult to prevent the formation of a boride, when a mixture of iron oxide and carbon is exposed to a temperature sufficient to melt the cast-iron thus produced, provided a fusible borate is used as a flux. Again, borax being such a desirable fluxing agent for metallurgical reductions, this compound is generally made use of in smelting small quantities of iron ores, for the purpose of further examination of the iron thus obtained. This reaction is, however, perfectly useless, provided a fluxable borate has been employed, as one hundred samples thus reduced by the author all contained more or less boron, averaging up to 2 per cent, while at the same time the iron in most cases appears solely as white cast, the carbon existing in the combined form; also, as a verification test, samples of cast-iron were exposed to different temperatures in common with fusible borates, the analyses of all these samples showing a deficiency on the total averaging from $\frac{1}{2}$ to 2 per cent.

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VOLUMETRIC DETERMINATION OF MOLYBDENUM AND VANADIUM.*

By CARL FRIEDHEIM.

IN concert with H. Euler I have elaborated a volumetric method for determining molybdenum. It consists in decomposing the molybdate or molybdenum tetroxide in Bunsen's apparatus with potassium iodide and hydrochloric acid, absorbing the liberated iodine in potassium iodide, and titrating with sodium thiosulphate in the usual manner.

F. A. Gooch and Charlotte Fairbanks (*Zeit. Anorg. Chem.*) have submitted this process to a re-examination, with the result that satisfactory values can be obtained only under conditions which they have ascertained and communicated.

Strictly speaking, it might be sufficient to point out that (as it appears from the figures given in my former

communication), in my method the errors fluctuate between -0.09 and $+0.36$ (and not as Gooch and Fairbank state, between $+0.05$ and 1.13 per cent), whilst the authors just named obtain errors of from -0.14 to $+0.45$, in order to prove that, according to the proposed modification, the results obtained are not better, but worse. But as my method admits of more general application, and on this account any additional difficulty must be avoided such as would be involved in the new proposals, I must examine more closely some of the declarations of Gooch and Fairbanks.

The hydriodic acid liberated on treating molybdenum tetroxide with potassium iodide and hydriodic acid is naturally—in as far as it is not oxidised by the molybdenum tetroxide—decomposed by the oxygen of the atmosphere, which naturally must signify an increased elimination of iodine. This I pointed out expressly in my former communication, in which it is stated:—"We have found that this process of determination can be carried out—with the due observation of certain precautions—much more rapidly and quite as accurately in Bunsen's apparatus, and therefore by distilling the substance with potassium iodide and hydrochloric acid."

But if we heat the mixture of the above substances rapidly, too much iodine is liberated in the receiver; but if we work in such a manner that the reduction of the substance is effected before hydriodic acid escapes, we obtain the theoretical quantity of iodine.

Hence the following *modus operandi* results for the execution of the method:— 0.2 to 0.3 gm. of the molybdate are mixed in the decomposition flask of Bunsen apparatus with 0.5 to 0.75 gm. of potassium iodide, and so much hydrochloric acid of sp. gr. 1.12 that the liquid may fill two-thirds of the flask. After connection with the escape tube and its introduction into the receiver, the contents of the flask are very slowly heated so as to reach ebullition, when the escape tube is filled as far as possible with iodine vapour and is just on the point of reflux. When the iodine is completely expelled, *i.e.*, when no more violet fumes are visible, and the solution takes a light-green colour, the distillation is at once broken off, and the iodine absorbed by potassium iodide in the receiver is titrated with sodium thiosulphate.

When Gooch and Fairbanks repeat these directions with the words "that the flask is two-thirds filled," and continue "the solution is not raised to ebullition until the flask is entirely filled with the heavy vapours of iodine, and is continued until iodine is no longer visible, and the liquid has taken a light-green colour," they have evidently quite overlooked that I laid particular emphasis on the exclusion of air; that is, on the non-contact of air and hydriodic acid.

(To be continued).

THE DETERMINATION OF ATOMIC MASSES OF SILVER, MERCURY, AND CADMIUM, BY THE ELECTROLYTIC METHOD.*

By WILLETT LEPLEY HARDIN.

(Continued from p. 79).

PART III.

Determination of the Atomic Mass of Cadmium.

NINE experimenters have determined the atomic mass of cadmium by many different methods, but the large variations in the results given by different chemists leave the true value of this constant still uncertain.

Stromeyer (Berzelius' "Lehrbuch," 5th Edition, iii., 1219) gave no details of his method of operation, but

* Contribution from the John Harrison Laboratory of Chemistry No. 13. From the author's thesis presented to the Faculty of the University of Pennsylvania for the degree of Ph.D.—From the *Journal of the American Chemical Society*, xviii., p. 990.

* *Berichte d. Chem. Gesell.*

found that 100 parts of cadmium combined with 14,352 parts of oxygen. On the basis of $O=16$, this ratio gives 111.483 for the atomic mass of cadmium. This result is much lower than those obtained by other experimenters, and is perhaps only of historical interest.

In a series of nine experiments, Von Hauer (*Journ. Prakt. Chem.*, lxxii., 350) determined the ratio of cadmium sulphate to cadmium sulphide. The sulphate used was purified by repeated re-crystallisations, and was finally dried at a temperature of 200° . After weighing the sulphate was always dried a second time and re-weighed. The two weighings never differed as much as one m.grm. The sulphide obtained was in each case tested for sulphate. The reduction of the sulphate to sulphide was accomplished by heating the sulphate in a current of dry hydrogen sulphide under pressure. The mean of nine observations computed on the basis of $O=16$ and $S=32.06$ gives 111.93 for the atomic mass of cadmium. Considering the large quantity of material used each time, and the precautions taken to insure accuracy, there seems to be little objection to the method.

Dumas (*Ann. Chim. Phys.*, [3], lv., 158) determined the ratio of cadmium chloride to metallic silver by titrating a solution containing a weighed quantity of cadmium chloride with a silver nitrate solution of known strength. The cadmium chloride was prepared by dissolving metallic cadmium in boiling hydrochloric acid. The solution was evaporated to dryness, and the chloride fused for six hours in a current of hydrochloric acid gas. The mean of six determinations gives 112.24 for the atomic mass of cadmium ($O=16$).

Maximum result, $Cd = 112.759$
Minimum ,, $Cd = 111.756$

Difference = 1.003

This large variation in the results obtained indicates the presence of impurities in the material used. In the first three experiments the cadmium was not purified; the mean of these three is $Cd=112.476$. The metal used in the last three experiments was considered by Dumas to be absolutely pure; the mean of the last three results is $Cd=112.007$. From the degree of purity of the cadmium chloride used in the different experiments, Dumas was inclined to reject the higher results, and concluded that the true atomic mass of cadmium was about 112.

Lensen (*Journ. Prakt. Chem.*, lxxix., 281) prepared pure cadmium oxalate by precipitating a solution of cadmium chloride, purified by repeated crystallisation, with pure oxalic acid. The precipitate was washed and carefully dried at a temperature of 150° . The mean of three results obtained by converting a weighed portion of the oxalate to oxide gives 112.06 for the atomic mass of cadmium ($O=16$). The small quantity of material used in the different experiments is somewhat objectionable.

Huntington (*Proc. Amer. Acad.*, xvii., 28) under the direction of Cooke, determined the ratio of cadmium bromide to silver bromide, and also the ratio of cadmium bromide to metallic silver. The bromide used was prepared by dissolving cadmium carbonate, which had been carefully purified, in pure hydrobromic acid. The product obtained was dried at a temperature of 200° , and finally sublimed in a porcelain tube in a current of dry carbon dioxide. In the first series of experiments the silver bromide corresponding to the cadmium bromide used was weighed. The mean of eight determinations computed from the total quantity of material used and silver bromide obtained, on the basis of $Ag=107.93$ and $Br=79.95$, is $Cd=112.24$. In the second series of experiments the quantity of metallic silver required to precipitate a known quantity of cadmium bromide was determined. The mean of eight determinations computed as in the first series gives 112.245 for the atomic mass of cadmium. The separate determinations in both series agree very closely.

Partridge (*Amer. Journ. Sci.*, [3], xl., 377) made three series of determinations. The first depended upon the conversion of cadmium oxalate into oxide, the second, on the reduction of the sulphate to sulphide, and the third, on the conversion of the oxalate into sulphide. The cadmium used in these experiments was purified by distilling twice *in vacuo*. Ten observations on the conversion of the oxalate into oxide, computed on the basis of $O=16$ and $C=12$, give 111.801 as a mean for the atomic mass of cadmium. Re-calculated by Clarke (*Amer. Chem. Journ.*, xiii., 34), on the basis of $O=16$ and $C=12.005$, the atomic mass of cadmium becomes 111.818. The mean of ten results obtained by reducing the sulphate to sulphide, computed on the basis of $O=16$ and $S=32$, gives 111.797 for the atomic mass of cadmium. Re-calculated by Clarke on the basis of $O=16$ and $S=32.074$, the atomic mass of cadmium is 111.711. In the third series the oxalate of cadmium was converted into sulphide by heating in a current of dry hydrogen sulphide. The mean of ten determinations, computed on the basis of $O=16$ and $S=32$, gives 111.805 for the atomic mass of cadmium. Re-calculated by Clarke on the basis of $O=16$ and $S=32.074$, the mean becomes 111.589. Partridge gives 111.8 for the atomic mass of cadmium, as a mean of the three series. If the higher values for carbon and sulphur be introduced this value becomes somewhat lower.

Jones (*Amer. Chem. Journ.*, xiv., 261) determined the atomic mass of cadmium by two different methods. The first was based on the conversion of the metal into oxide, and the second on the conversion of the oxalate into oxide. The cadmium used was distilled six times *in vacuo*. The last distillate was tested spectroscopically, and found to be free from impurities. In the first series of experiments a weighed portion of the pure metal was dissolved in pure nitric acid in a porcelain crucible. The solution was evaporated to dryness, and the resulting cadmium nitrate ignited to oxide. The final decomposition was accomplished by means of a blast lamp. Reducing gases were carefully excluded from the crucible during the process of ignition. The weighings were all made against a tared crucible. The mean of ten observations, computed on a basis of $O=16$, gives 112.07 for the atomic mass of cadmium. The different determinations agree very closely. In the second series of experiments cadmium oxalate, prepared by precipitating pure cadmium nitrate with pure oxalic acid, was converted into oxide. The material was carefully ignited until the oxalate was decomposed; it was then treated with nitric acid, and again ignited in a manner similar to that described in the first series. The mean of five determinations computed on the basis of $O=16$ and $C=12.003$, is $Cd=111.032$. From all the observations Jones concludes that 112.07 represents very closely the atomic mass of cadmium ($O=16$).

Lorimer and Smith (*Ztschr. Anorg. Chem.*, i., 364) determined the ratio of the atomic mass of cadmium to that of oxygen by dissolving pure cadmium oxide in potassium cyanide and electrolyzing the solution. To obtain pure material, the commercial cadmium was dissolved in nitric acid, and the solution evaporated to crystallisation. The crystals of cadmium nitrate were removed from the liquid, dissolved in pure water, and re-crystallised. The product obtained by the second re-crystallisation was dissolved in a little water and treated with a slight excess of potassium cyanide in a platinum dish. From this solution the metallic cadmium was thrown out by means of the electric current. The nitrate obtained by dissolving the electrolytic cadmium in pure nitric acid was tested spectroscopically and found to be free from impurities. The pure cadmium nitrate was digested with ammonium hydroxide and ammonium carbonate, and the resulting cadmium carbonate ignited to oxide in a platinum crucible. The method of operation was very simple, a weighed portion of the oxide was dissolved in pure potassium cyanide, the solution electrolysed, and the resulting metallic cadmium weighed. The

mean of nine observations computed on the basis of $O=16$ gives 112.055 for the atomic mass of cadmium.

Bucher (Thesis, Johns Hopkins University, 1894) made six series of experiments. The cadmium used was purified by nine distillations *in vacuo*. The weighings were all reduced to a vacuum standard, and computed on the basis of $O=16$, $S=32.059$, $C=12.003$, $Cl=35.45$, $Br=79.95$, and $Ag=107.93$.

In the first series cadmium oxalate, dried for fifty hours at 150° , was ignited to oxide. The mean of eight observations gives 111.89 for the atomic mass of cadmium.

In the second series, cadmium oxalate was converted into sulphide by heating in a current of dry hydrogen sulphide. The mean of four determinations is $Cd=112.15$.

In the third series a weighed quantity of cadmium chloride, dried at a temperature of 300° in hydrochloric acid gas, was precipitated with silver nitrate, and the resulting silver chloride weighed. The mean of twenty-one determinations is $Cd=112.39$. The separate observations in this series agree very closely.

The fourth series was similar to the third, except that cadmium bromide was used instead of the chloride. The mean of five determinations is $Cd=112.38$, a result almost identical with that obtained from the chloride.

In the fifth series a weighed portion of metallic cadmium was converted into sulphate, which was dried at 400° and weighed. The excess of sulphuric acid which remained with the sulphate was estimated and its weight deducted. The only result given is $Cd=112.35$.

In the last series metallic cadmium was converted into oxide by dissolving in nitric acid and igniting the resulting cadmium nitrate. The mean of two determinations made by igniting the material in a porcelain crucible gives 112.08 for the atomic mass of cadmium. Three similar determinations made with a platinum crucible gave as a mean $Cd=111.87$. From a series of experiments on cadmium oxide, Bucher concluded that a correction should be applied to the last and also the first series. By making this correction, the results in these two series would be very close to those obtained from the chloride and bromide.

From all the preceding determinations Clarke gives 111.93 as the most probable value for the atomic mass of cadmium. The large variation in the results of different experimenters has not been fully explained. Some chemists think that the larger values are due to a higher degree of purity in the metallic cadmium used, and hence regard these values as being more nearly correct. But it must be remembered that the reverse is true in the experiments of Dumas. From material which had not been purified, Dumas obtained results ranging from 112.32 to 112.76 for the atomic mass of cadmium, while from material which he considered absolutely pure, the results were from 111.76 to 112.13 .

Preparation of Pure Cadmium.

The metallic cadmium used in these experiments was purified by distillation in a current of hydrogen which had been passed through solutions of caustic potash, lead nitrate, potassium permanganate, and sulphuric acid. A hard glass combustion tube was heated to redness, and the walls of the tube indented at two points with a three-cornered file. This divided the tube into three parts. Commercial cadmium was placed in one end of the tube, and connection made with the hydrogen generator. After complete removal of the air, the tube was carefully heated in a combustion furnace until one-half of the metal had distilled over into the middle portion of the tube. The metal was cooled in a current of hydrogen. The tube was then broken and the metal removed. The portions in the first and last sections of the tube were rejected. The middle portion was placed in a second combustion tube, similar to the first, and the distillation repeated. After three distillations the metal was

examined spectroscopically and found to be free from impurities.

(To be continued).

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Special General Meeting, February 12th, 1897.

THE Chair was taken by Captain ABNEY, who, as retiring President, referred to some of the changes which had occurred in the Society during the past year.

The Annual Subscription had been raised, but a satisfactory number of new Fellows had been enrolled. The Society had lost two by death. A good deal of work had been done in the direction suggested by the discoveries of Röntgen.

The Treasurer, Dr. ATKINSON, then presented his Report and Balance-sheet for the year 1896. There was evidence of improvement in the financial position, but there was still a deficiency to be met. Profits from sales of publications had been small; it was desirable to reduce the price of the volumes of collected papers of Joule and Wheatstone, and to call the attention of physicists to these valuable records of classical work.

Mr. WALKER suggested that physical laboratories, especially those in London, should be visited by Fellows of the Society, with a view to comparing notes as to the construction of apparatus; professors of colleges and other institutions should be invited to appoint visiting days for this purpose.

Votes of thanks were passed to the retiring President, Council, and Officers, and also to the Council of the Chemical Society for the use of their rooms at Burlington House.

In replying, Capt. ABNEY said that the coming year would probably bring about further improvements in the system of abstracting and indexing, by co-operation with other societies at home and abroad. He then read the list of Council and Officers for the year 1897-8:—

President—Shelford Bidwell, M.A., LL.B., F.R.S.

Vice-Presidents, who have filled the Office of President—Dr. Gladstone, Prof. G. C. Foster, Prof. Adams, The Lord Kelvin, Prof. Clifton, Prof. Reinold, Prof. Ayrton, Prof. Fitzgerald, Prof. Rücker, Capt. Abney.

Vice-Presidents—Maj.-Gen. E. R. Festing, L. Fletcher, Prof. Perry, G. Johnstone Stoney.

Secretaries—T. H. Blakesley, H. M. Elder.

Foreign Secretary (new office)—Prof. S. P. Thompson.

Treasurer—Dr. Atkinson.

Librarian—C. Vernon Boys.

Other Members of Council—Walter Bailey, L. Clark, A. H. Fison, Prof. Fleming, R. J. Glazebrook, Prof. A. Gray, G. Griffith, Prof. Minchin, Prof. Ramsay, J. Walker.

The newly-elected President, Mr. SHELFORD BIDWELL, then took the Chair, and an Ordinary Meeting was held.

Mr. BLAKESLEY read a paper by Mr. H. H. HOFFERT, "On the Use of very small Mirrors with Paraffin Lamp and Scale."

For the mirrors of reflecting instruments the author prefers small rectangular strips of microscope cover-glass, chosen thin and plane. These are first silvered, and then cut to shape by a splinter of diamond embedded in wax. They are about 8 m.m. long by 1.5 m.m. broad, and are suspended so that their longest sides are vertical. Rectangular mirrors suspended in this way are lighter, and have less inertia than round mirrors of equal aperture. A paraffin-lamp flame placed edgewise to the mirror gives sufficient illumination. The image of the flame is focussed on the mirror by a lens midway between them; it is a

right vertical line, and thus conforms to the shape of the mirror. A scale is fixed upon a screen between the lens and lamp; and the screen has a circular aperture just below the centre of the scale, provided with a vertical cross-wire. The relative position of screen and lens is adjusted so that an image of the wire is formed upon the scale after reflection at the mirror.

Mr. Boys said he had frequently used small mirrors constructed as described by the author, and he could not see what was new in the method, except that a paraffin lamp had been found sufficiently bright for the purpose. It is desirable to diminish inertia by choosing extremely thin glass. Microscope cover-glasses are generally supplied in squares or discs very fairly equal in size; if they are dealt out on a table, like a pack of cards, their relative thicknesses can be judged by the note produced as they fall. Flatness can be estimated nearly enough by balancing them one by one upon the knuckle, nearly level with the eye, and observing the reflection of an illuminated straight edge, such as a window-bar. All rejected glasses should be broken. The good ones can be further examined by a telescope and artificial star. A common writing diamond is best for cutting the thin plates. Special care must be taken not to distort the mirror in fixing to the suspended system. If liquid shellac is used in the attachment distortion will certainly occur,—at any rate, if it is applied throughout the whole length of the mirror. The best way is to make the attachment at a mere point, near the top of the mirror, using a speck of shellac as viscous as possible, and heating, if necessary, by radiation, not by conduction. Mr. Boys thought that a reflecting prism near the mirror might be used in certain cases where a paraffin lamp with its inevitable vertical flame was required for horizontal projections. For general purposes Mr. Boys prefers some such arrangement as the following:—If the source of light is a point, a lens is employed, forming an image of the source upon the mirror. (If the source of light is a surface, this lens is evidently superfluous). The cross-wire is stretched near to the lens on the side towards the mirror. It is now necessary to focus the cross-wire upon the scale, and this is best done by a plano-convex fixed lens as near as possible to the mirror, with its plane face towards the mirror. The light passes twice through the lens. As it may be necessary to change the plano-convex lens from time to time, according to the distance of the scale, Mr. Boys attaches it with a little vaseline to a strip of plate glass in front of the instrument. One advantage of such an optical system is that it allows the instrument to be set up in the same position with respect to the scale at all times.

Dr. THOMPSON pointed out that Mr. Hoffert had obtained his results using only *one* lens, by properly choosing the position of the cross-wire.

A vote of thanks was given to the author, and the meeting adjourned until February 26th.

NOTICES OF BOOKS.

Water and its Purification; a Handbook for the Use of Local Authorities, Sanitary Officers, and others interested in Water Supply. By SAMUEL RIDEAL, D.Sc., Fellow of University College, London, F.I.C.; Examiner in Chemistry to Royal College of Physicians; Public Analyst for the Lewisham District Board of Works; Water Examiner to the Guildford Rural District Council; Author of "Disinfection and Disinfectants." With numerous Illustrations and Tables. London: Crosby Lockwood and Son, Stationers' Hall Court. Pp. 292. 1897.

WRITINGS discussing municipal water supply resolve themselves into two grand categories. The one deals

with the outgoing waters, their purification or disinfection, and their utilisation. The other class, of which the volume before us is an excellent specimen, discusses the incoming waters, their suitability for domestic and industrial purposes, and their improvement whenever practicable.

In the remarks on natural waters Dr. Rideal shows that an appeal to the physical characters of a water is not trustworthy, and may even be dangerously deceptive. He rightly urges that even traces of the poisonous metals render a water inadmissible for household consumption. Lead, the most insidious and dangerous of metallic impurities, is, unfortunately, often introduced by lead service-pipes. Some waters, however, have the dangerous property of rapidly attacking lead. This is especially the case with peaty waters. Cisterns for water-supply are rightly and emphatically condemned.

It is an unfortunate circumstance that the upland areas, uncultivated and unpeopled, are not sufficient to afford drainage grounds for the supply of adjacent cities. The instance of Manchester is most instructive. A few years ago its supply from a series of reservoirs constructed in Longdendale (not Longerdale), the drainage off the mill-stone grit, and the uncultivated moor-lands, was viewed as sufficient not merely for the present but the future. But recently it has been found insufficient, and an additional supply has been obtained from Thirlmere in Cumberland. Contrary to our fears the engineering operations near this lake have not interfered with the beauty of the scenery.

Dr. Rideal rightly holds that it is the duty of the State to apportion the upland water-sources to the needs of the population.

Mention is made of the Holmfirth catastrophe, and of the still more appalling disaster at Sheffield.

The London County Council proposes to derive the future supply of the metropolis from Wales, taking the head waters of the Usk, Wye, and Towey, and conveying them by two aqueducts of respectively 150 and 175 miles in length. The great objection to this scheme would be the necessity of guarding the aqueducts and bridges from possible mischief on the part of bodies whom—as we are not a political organ—we cannot here point out. The suggestion that the purer mountain water might be "exclusively used for drinking purposes," and employing ordinary waters for washing, trade, and sanitary purposes, would certainly lessen the demand on the upland areas, but would at the same time burden the consumer with the expense of a duplicate system of mains and service pipes.

On page 101 we find the standards of the Thames Conservancy. This body demands that discharges into the river must be:—

1. Free from offensive odour.
2. Free from suspended matter.
3. Neither acid or alkaline to test papers.

This requirement Dr. Rideal acutely pronounces impossible, since natural waters are almost invariably acid to one test on account of free carbonic acid, and alkaline to another owing to carbonates. The author suggests that limits of permissibility should be given.

4. Not more than 60 grs. per gallon of total solids.
5. Not more than 2 grs. per gallon of organic carbon, and 0.75 grs. per gallon of organic and ammoniacal nitrogen.
6. Not less than one cubic inch of free oxygen per gallon.

Concerning the action of soft waters upon leads we find two conflicting statements. On page 117 we read "that its (the water of Loch Katrine) only fault is that it acts rapidly upon lead, of which we shall speak further." But on page 137, after speaking of the soft water of the Varty (Dublin), the author writes: "The equally soft water of Loch Katrine has little or no action upon this metal."

On page 97 Dr. Rideal points out the danger of allowing pigs to wade in streams, and consequently to introduce into them parasites.

The self-purification of rivers is exemplified in Dr. P. Frankland's paper on the river Dee (p. 105).

On the question of constant *v.* intermittent water service the author takes the right view, condemning the latter procedure, which has justly gone out of use in most places except London.

In speaking of filters, which the author does at great length and with thorough fairness and accuracy, he shows that all domestic filters, except the Pasteur-Chamberland, are of very doubtful efficacy, if not positively harmful. The Berkenfeld filter requires to be boiled either once or twice every twenty-four hours.

Referring to the mains and service pipes for the distribution of water in cities, our author recommends that the pipes should be laid not less than four feet below the surface, and should pass within about three feet of the kitchen grate before branching off to different parts of the establishment.

It is unfortunate that no one has the power to enforce these standards as against "Jerry."

In our opinion Dr. Rideal has made in this book a valuable contribution to our sanitary literature.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 3, January 18, 1897.

Researches on Helium.—M. Berthelot.—The author has succeeded in combining helium, both with the elements of the hydrogen carbides, and with those of carbon disulphide, with the intervention of mercury and the influence of the electric effluve, all precisely under the same conditions in which he has realised the combination of argon. In both cases these syntheses have been checked by analysis; that is to say, by the regeneration of the element in a free state.

Remarks on the Specific Heats of Elementary Gases, and on their Atomic Constitution.—M. Berthelot.—The author calls attention to the following values of specific heat at constant volumes, referred to an identical volume of the elements, such as that occupied by 2 grms. of hydrogen:—

Gas with a monatomic mol.	3°0
Diatomic gas	4°8 (not split up at present, and 6°6 split up above 1500°).

Tetatomic gases	11°4
---------------------------	------

It will be remarked that the ratio of these values, measured near the ordinary temperature (between 0° and 300°), is not remote from that of 1 : 2 : 4; that is to say, that the specific heats of the simple gases at a constant volume are approximately proportional to the number of atoms contained in the molecule. Besides the four groups of gaseous elements just distinguished, there are others, such as contain triatomic elements, *e.g.*, ozone, comparable to hyponitric gas; and certain hexatomic elements, sulphur and selenium. But their specific heat is hitherto unknown. We know how for the simplified notions deduced from a first study of the elementary gases anciently known, *e.g.*, hydrogen, oxygen, and nitrogen, there are being substituted more profound notions on the physico-chemical constitution of the elements.

Classification of the Chemical Elements.—Lecoq de Boisbaudran.—This paper will be inserted at some length.

Certain Salts and Derivatives of Dinitro-ortho-cresol.—P. Cazeneuve.

Law of the Transparency of Gases for X Rays.—L. Benoist.—In the earliest experiments on the X rays it was remarked that the opacity of different bodies for the rays increased in general with their density, and it was thought that a simple relation such as direct proportionality might exist between these two characters. With gaseous bodies the case is different. With sulphurous acid, methyl chloride, and air the absorption produced is proportional to the density of the gas employed. This is the relation which Lenard has already obtained for the cathodic rays before Röntgen's discovery. As the specific absorbent power of gases resulting from the foregoing measurements gives the value 0·14, aluminium gives the value 0·09; the deviation is not great, whence aluminium would almost satisfy a general law of proportionality between the absorbent power and the density. On the contrary, silver gave 0·84—a number six times too great.

Speed of the Reduction of Chromic Acid by Phosphorous Acid.—G. Viard.—This paper requires the formulæ and the table here introduced.

Action of Hydrogen Sulphide and Selenide upon Phosphonyl Chloride.—A. Besson.—If we dissolve dry hydrogen sulphide at 0° in POCl₃ and leave it in a closed vessel, in a few hours the liquid becomes milky, and after twenty-four hours there collects at the lower part of the vessel a light bulky amorphous precipitate, whilst the supernatant liquid becomes limpid. If the experiment is prolonged, there is a slow formation of crystalline needles. Both the amorphous precipitate and the crystalline needles have the same composition, P₂O₂S₃; that is, an oxy-sulphide. Dry hydrogen selenide has no perceptible action upon POCl₃ in the cold, but at 100° it reacts slowly, forming phosphonyl selenide.

Action of Ethylalyl Chloride upon Pseudocumene and Mesitylene.—E. Bouveault.—These two papers are not suitable for abridgment.

Decrease of the Nitrogenous Matter in the Wheats of the Department "Du Nord."—M. Balland.—The nitrogenous matter, which in 1848 varied between 10·23 and 13·02 per cent, is now between 8·96 and 10·62 per cent.

The Reproduction of Colour by Photographic Methods.—Sir Henry Trueman Wood is announced to read a paper at the Society of Arts on this subject on the 24th inst. Captain Abney, C.B., F.R.S., will preside. Illustrations will be shown of several of the recently invented processes for the photographic reproduction of colour.

On Chromium and Manganese Phosphides.—A. Granger.—The analysis of these phosphides is delicate, on account of the difficulty of separating phosphoric acid from chromium and manganese oxides. The author reduced the substances to a fine powder, treated them with melting potassa, raising the temperature gradually to dull redness, and keeping the mass in tranquil fusion for 1½ hours. The mass, when cold, is dissolved in boiling water, acidified with nitric acid in the case of chrome, or with hydrochloric acid in the case of manganese. The chromic solution is then treated with ammonium molybdate to precipitate phosphoric acid. In the filtrate the chlorate is reduced to the state of a salt of chrome which is precipitated in the state of sesquioxide, carrying molybdenum down with it. The precipitate, dissolved and re-precipitated, yields an oxide sufficiently free from foreign salts. The manganic solution, evaporated to dryness with hydrochloric acid in excess, is neutralised with ammonia, and ammonium sulphide is added. We evaporate to dryness, and add a further quantity of the latter reagent; re-dissolved in water, and filtered, the precipitate contains all the manganese, and in the filtrate the phosphoric acid is determined as usual.

MEETINGS FOR THE WEEK.

- MONDAY, 22nd.**—Society of Arts, 8. (Cantor Lectures). "Industrial Uses of Cellulose," by C. F. Cross, F.C.S.
- TUESDAY, 23rd.**—Royal Institution, 3. "Animal Electricity," by Prof. A. D. Waller, F.R.S.
- WEDNESDAY, 24th.**—Society of Arts, 8. "Reproduction of Colour by Photographic Methods," by Sir Henry Truman Wood.
- THURSDAY, 25th.**—Royal Institution, 3. "The Problems of Arctic Geology," by J. W. Gregory, D.Sc., F.R.S.
- Society of Arts, 8. "The Mechanical Production of Cold," by Prof. James A. Ewing, M.A., F.R.S.
- FRIDAY, 26th.**—Royal Institution, 9. "Palestine Exploration," by Lieut. Col. C. R. Conder, R.E.
- Physical, 5. "Photography of Ripples," by J. H. Vincent.
- SATURDAY, 27th.**—Royal Institution, 3. "The Growth of the Mediterranean Route to the East," by Walter Frewen Lord.

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THE CHEMICAL NEWS.

Vol. LXXV., No. 1944.

ON THE REMOVAL OF OXIDE FROM MELTED COPPER AND COPPER ALLOYS.

By SERGIUS KERN, M.E., St. Petersburg.

THE following is perhaps not new to many metallurgists, but the *modus operandi* of the application of phosphorus for cleaning copper alloys, just before casting them into ingots or mouldings, must be new to many.

In order to reduce the oxides and to obtain dense and solid castings, for several years the Obouchoff Works here have successfully used our plan, which is as follows:—

In order to safely throw the phosphorus into the pots of melted copper, just before drawing them out of the furnace, the phosphorus is submitted to the following treatment:—Phosphorus is cut into pieces, weighing about 3 ounces each, and placed in a concentrated solution of copper sulphate, where it is kept for further use. From time to time fresh copper sulphate is added, and pieces of phosphorus also, after taking out for foundry uses pieces of phosphorus already covered with precipitated copper.

In this manner it is easy to handle phosphorus, which also when thrown into pots does not take fire at once, but safely reaches the melted metal to act in the desired manner.

About two pieces are usually sufficient for 1 cwt. of metal in the pot.

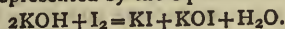
The addition of phosphorus in the mentioned manner to copper plays the part of ferromanganese to steel.

February 13, 1897.

HYPOIOUS ACID AND HYPOIODITES.*

By R. L. TAYLOR, F.C.S.

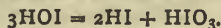
THE author confirms and extends the observations of Schönbein, using, as he did, an aqueous solution of iodine. When a little alkali is added there is practically no iodate formed, from 90 to 95 per cent of the iodine undergoing the reaction represented by the equation—



This is proved by the bleaching action upon a standard solution of indigo, and also by Schwicker's method of decomposing the hypoiodite by a bicarbonate or by carbonic acid.

The solution bleaches indigo much more rapidly than chlorine or hypochlorites, but does not bleach litmus. It gives a precipitate with cobalt nitrate which blackens on standing, and an immediate brown precipitate with manganese salts and lead salts. Stronger solutions are obtained by using iodine water containing a little suspended iodine. All the solutions are decomposed completely by boiling for three or four minutes. The author concludes that the reaction represented above, as Walker and Ray have also recently stated, is a balanced one. He has repeated Lunge and Schoch's experiments, who obtained, by the action of iodine upon lime in presence of comparatively little water, a bleaching liquid which stood being boiled for hours without being decomposed. He

concludes that their observations were practically only correct so far as they agreed with those of Schönbein. The author has also investigated the action of precipitated mercuric oxide upon iodine water, and find that hypoiodous acid is formed. The filtered (colourless) solution possesses only the feeblest possible bleaching properties, but the addition of a little alkali transforms it at once into as strong a bleaching solution as Schönbein's, and which it now exactly resembles. By the methods mentioned above it is found that from 40 to 45 per cent, out of a possible 50 per cent, of the iodine used exists in the solution as hypoiodous acid. By using iodine water and a little suspended iodine, a stronger solution is obtained, which very soon decomposes, turning brown, owing to the liberation of iodine. The hypoiodous acid probably decomposes as follows:—



and the hydriodic acid and iodic acid immediately decompose each other, with liberation of iodine. The author suggests a possible explanation of the feeble bleaching power of the free acid in the instability of the hydriodic acid which will be left, compared with the alkaline iodides.

A solution containing hypoiodous acid also appears to be formed by the action of various silver salts upon iodine water, the best results being obtained with silver nitrate and silver carbonate. These liquids bleach much more rapidly than the one described above, probably because of the silver which is present; but they are much less rapid in their bleaching action than the alkaline hypoiodites. In other respects they resemble Schönbein's solutions, being, however, much less stable, this again being probably due to the presence of silver in the solution. The liquid obtained by adding a few drops of nitrate of silver to aqueous iodine loses 90 per cent of its bleaching power in five minutes.

OPENING UP OF SILICATES.

By P. JANNASCH.

FOR this purpose the author uses pure lead silicate, and finds it satisfactory. He proceeded from Bong's proposal to use lead oxide for this purpose, supported upon the favourable experiments which he made in concert with J. Locke in determining the water in topaz. Jannasch proposes to mix the silicate (freed from organic matter, if necessary by ignition) with from ten to twelve parts of lead carbonate, and heat it in a platinum crucible, 52 to 53 m.m. in height, and 45 m.m. wide at top, for from fifteen to twenty minutes at first, with a flame of about an inch in height. It is then heated to fusion, but the crucible must not be red-hot for more than one-third of its height. The fusion is continued for ten to fifteen minutes. Care must be taken that the flame does not smoke in order to avoid any reductive action. The crucible is taken directly from the flame and plunged into cold distilled water. The melt is removed from the crucible by gentle tapping and squeezing, and decomposed with nitric acid, and finally evaporated repeatedly to dryness with concentrated nitric acid, and the residue is taken up with 10 c.c. of concentrated nitric acid, diluted with 75 to 100 c.c. of water, heated on the water-bath for fifteen minutes, filtered and washed.

The filtrate is mixed with abundance of hydrochloric acid; after settling the lead chloride is sucked off and washed with hydrochloric acid (1 vol. concentrated acid + 1 vol. water). The filtrate is evaporated down (to expel nitric acid) with 30 c.c. hydrochloric acid (1 : 4) and as much water. The residue of the lead chloride is filtered off and quickly washed with cold water. The traces of lead left in the filtrate are precipitated with sulphuretted hydrogen. The hydrogen sulphide is then entirely removed from the filtrate.—*Zeit. Anorg. Chemie*.

* Summary of a Paper read at the Manchester Literary and Philosophical Society, February 9th, 1897.

A SENSITIVE SIMPLE REACTION FOR
NITROUS ACID.

By Prof. Dr. E. RIEGLER.

We introduce into a small test-tube about 2 or 3 centigrams. of crystallised naphthionic acid, and from 5 to 6 c.c. of the liquid to be examined for nitrous acid, shake up well, add 2 or 3 drops of concentrated hydrochloric acid, shake up thoroughly for a minute, and allow from 20 to 30 drops of ammonia to flow slowly into the test-tube whilst held in a slanting position; when there appears at the surface of contact of the liquids a rose-coloured ring, even if only traces of nitrous acid are present. If we shake up the entire liquid it becomes rose-coloured or deep red, according to the quantity of the nitrous acid.

As very dilute solutions of naphthionic acid have a violet-blue fluorescence, it is advantageous to examine the colour by transmitted light.

The reaction consists herein, that the naphthionic acid is converted by the nitrous acid into diazo-naphthalin sulphonic acid, which forms with another mol. of naphthionic acid, and with ammonia a colouring matter which determines the rose colouration.

In rain and drinking waters the nitrous acid can be very finely shown by means of this reaction. The detection of nitrites in saliva is extremely instructive. To this end we dilute the saliva with five times its volume of pure distilled water, filter, and treat 5 or 6 c.c. of the filtrate as above directed.

Traces of nitrous acid may in like manner be detectable in urine.—*Zeit. Analyt. Chemie.*

WORKING UP URANIUM RESIDUES.

By A. GOWALOWSKI.

In this paper I formerly described a method for working up uranium phosphate residues. Supported upon Laugier's observations I used ammonium carbonate for dissolving hydrated uranium phosphate, but I have now abandoned this method in favour of sodium carbonate (ammonia soda), which is both cheaper and has a greater solvent power. By introducing the uranic precipitate into a strong solution of ammonia soda, I dissolve the uranium phosphate, filter, and add rather more ferric chloride than is necessary to take up all the phosphoric acid, and filter again, when iron phosphate and ferric hydrate remain, which are washed with water. I treat the entire filtrate in small portions with magnesia mixture as long as there appears a turbidity of ammonio-magnesium phosphate, whereby any residues of phosphoric acid are separated. From the solution precipitate uranium oxide with sodium carbonate; filter after standing for twenty-four hours, and dissolve either at once in acetic acid or acidify previously with hydrochloric acid, boil to expel carbonic acid, precipitate with ammonia, collect the ammonia-uranic oxide on the filter, wash, and dissolve in acetic acid or in nitric or hydrochloric acid.—*Zeit. Analyt. Chemie.*

Isomerism of Structure and of Rotatory Power.—Ph. A. Guye and J. Guechgorine.—It results from these researches that we know at present three series of propylic isomers and three series of butylic isomers among the derivatives of active amylic alcohol. If we take account of the decreasing character of the rotatory powers in each of these series, we conclude that in all these series the propylic group behaves as heavier than the isopropyl group, and that the isobutyl group behaves as heavier than normal butyl, which in turn acts as heavier than secondary butyl.—*Comptes Rendus*, cxxiv., No. 5.

ON THE ACTION OF WAGNER'S REAGENT
UPON CAFFEINE, AND
A NEW METHOD FOR THE ESTIMATION
OF CAFFEINE.*

By M. GOMBERG.

(Concluded from p. 90).

Estimation of Caffeine.

ALL the methods for the estimation of caffeine depend upon the extraction of the alkaloid by an immiscible solvent from either a dry residue or from its solution in water. But Spencer (*Fourm. Anal. Chem.*, iv., 390, 1890) has recently shown how difficult it is to remove the alkaloid from its solution in water. According to him, it is necessary to shake out the liquid at least seven times with chloroform, in order to remove caffeine quantitatively. It is usually stated that caffeine does not form any stable salts in a watery solution, and consequently it can be shaken out with immiscible solvents from either alkaline or acid solutions. But this is only relatively true, as will appear from the following illustrations. 1.0085 grms. of caffeine were dissolved in 60 c.c. of sulphuric acid (1:10), and this solution was repeatedly shaken out with chloroform, 25 c.c. at a time.

10 consecutive portions of chloroform gave a total of	0.3514 grm. caffeine.
3 additional portions of chloroform made a total of	0.4859 " "
3 more additional portions of chloroform made a total of . .	0.5034 " "

The extreme delicacy of the test for caffeine by means of Wagner's reagent has suggested the possibility of applying this reagent for the quantitative estimation of the alkaloid. Its successful application necessitates, of course, a solution of the alkaloid free from other substances that are precipitated by, or absorb iodine,—a condition requisite in the estimation of any base by means of Wagner's reagent. This method gives very satisfactory results, as nearly theoretical as could be expected. I am indebted for the analytical data of the subjoined table to Mr. James W. Knox, holder of the Stearns' Fellowship in the School of Pharmacy. The method of procedure employed by us was practically the same as that used by Kippenberger. Definite volumes of acidulated solutions of caffeine were precipitated with a known volume of iodine in potassium iodide. After complete precipitation an aliquot portion of the supernatant liquid was obtained, either by decantation or filtration, and the excess of iodine was estimated by titrating against a tenth normal solution of sodium thiosulphate. The precipitation is best performed in a tall test-tube on foot, and the solution for titration is removed directly by immersing the end of the burette into the liquid and applying suction at the upper end. When it is desirable to filter off an aliquot portion, a filter of glass-wool and asbestos gives very satisfactory results.

We have tested the method on solutions of caffeine acidulated with sulphuric acid, the solutions being of different strengths, namely, containing 0.25 per cent of caffeine, 0.50 per cent, 0.75 per cent, and 1.00 per cent respectively. We have varied in different series of experiments the amounts of Wagner's reagent, employing just the theoretical quantities, a small and large excess above that, as well as quantities below those required by the theory. Columns I., II., III., and IV., give the results obtained by allowing the solutions to stand for an hour before decanting an aliquot portion for titration; column V. shows the results obtained when the liquid for titration was filtered off within five minutes after the addition of Wagner's reagent. The results are calculated on the basis that the periodide has the composition

* From the *Journal of the American Chemical Society*, xviii., No. 4.

Wagner's reagent employed.	Solution containing 0.25 per cent of caffeine.			Solution containing 0.50 per cent of caffeine.			Solution containing 0.75 per cent of caffeine.			Solution containing 1.00 per cent of caffeine.			Solution containing 0.50 per cent of caffeine.		
	Taken.	Found.	Per cent recovered.	Taken.	Found.	Per cent recovered.	Taken.	Found.	Per cent recovered.	Taken.	Found.	Per cent recovered.	Taken.	Found.	Per cent recovered.
Theoretical quantity + 2 c.c.	0.0600	0.0591	98.33	0.1200	0.1175	97.82	0.1500	0.1481	98.40	0.1200	0.1182	98.38	0.1200	0.1154	96.78
	0.0589			0.1175			0.1471			0.1179			0.1165		
if theoretical quantity ..	0.0750	0.0749	99.88	0.1191	0.1189	99.17	0.1489	0.1485	99.13	0.1200	0.1191	99.12	0.1200	0.1196	99.75
	0.0749			0.1189			0.1485			0.1187			0.1197		
Twice the theoretical quantity ..	0.0500	0.0502	100.80	0.0805	0.0805	99.63	0.1200	0.1184	98.67	0.1100	0.1091	99.18	0.0800	0.0802	99.63
	0.0506			0.0789						0.1091			0.0791		
One-half of the theoretical quantity ..	0.0750	0.0363	48.40	0.1500	0.0794	49.62	0.2250	0.1107	49.20	0.2000	0.1067	53.35	0.1500	0.0791	49.44
	0.0363			0.0794			0.1107			0.1067			0.0791		

$C_8H_{10}N_4O_2.HI.I_4$. The amount of alkaloid is calculated from the amount of iodine used up, by the formula—

$$4I : C_8H_{10}N_4O_2 :: 506 : 194;$$

i.e., one part of iodine represents 0.3834 part of caffeine. Or, 1 c.c. tenth normal iodine = 0.00485 grm. caffeine.

The results presented below show that the estimation of caffeine by this method is very exact. The best results are obtained when iodine is in considerable excess, as is evident from the figures obtained where one and one-third and twice the theoretical quantities of Wagner's reagent were used. All the results in the table were obtained on solutions of caffeine acidulated with sulphuric acid, the acidulation being tolerably strong, about 1 c.c. of the concentrated acid to 50 c.c. of the liquid. Experiments upon the influence of the acid indicate that a large excess of sulphuric acid interferes to some extent with the reaction. The amount of recovered caffeine falls as low as 95 per cent of the quantity taken, when 4 c.c. of the concentrated acid to 50 c.c. of the liquid are used. The results are also not very uniform and concordant. The fact that the precipitation of caffeine by Wagner's reagent is more delicate in presence of hydrochloric acid than any other acid would make it advisable to employ that acid in quantitative estimation of the base by iodine.

This method could easily be employed for the estimation of the alkaloid in caffeine-bearing drugs. Of course, it is necessary to have the final solution of the alkaloid in water as free as possible from other substances that may be precipitated by Wagner's reagent. The estimation of caffeine by this method is likely to give higher results than have hitherto been obtained. The following procedure is recommended:—"The drug is thoroughly digested with water for some time, by the aid of heat, cooled, and made up to a definite volume, and filtered. An aliquot portion of the filtrate is treated with lead acetate, the precipitate allowed to settle and filtered. The whole of the filtrate, or a given portion of it, is treated with hydrogen sulphide to remove the lead, and filtered. This filtrate, after boiling off the hydrogen sulphide, is divided into two equal portions, and each treated with a definite volume of the standard iodine solution,—the first portion without the addition of any mineral acid, the second with the addition of hydrochloric or sulphuric acid. After five to ten minutes' standing the excess of iodine is estimated in each of the two solutions, as described above. The first portion, containing no other but some acetic acid, serves to indicate whether the filtrate from the lead sulphide contains any other materials besides caffeine that are likely to be precipitated by Wagner's reagent,—for caffeine itself is not precipitated by it even in presence of tolerably strong acetic acid. If any absorption of iodine be found in the first portion, then that quantity is to be subtracted from the amount of iodine taken up by the second portion; the difference represents the iodine used up in the formation of the periodide of caffeine. The amount thus used up multiplied by 0.3834 gives the amount of caffeine in that particular portion of the liquid.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING JANUARY 31ST, 1897.

By WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, *Metropolis Water Act*, 1871.

London, February 11th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 182 samples

* These directions are in part given by Spencer, 1890 (*Journ. Anal. Chem.*, iv., 390).

of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Jan. 1st to Jan. 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in a previous report.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 182 samples examined all were found to be clear, bright, and well filtered.

The rainfall at Oxford during the month still shows a small deficiency, the average fall for the last thirty years is 2.16 inches, the actual fall in January was 1.85 inches, making a deficiency of 0.31 inch; it has been pretty fairly distributed through the month, the greatest fall being 0.58 inch on the 8th.

Our bacteriological examinations have been, and will in future be, conducted on a much larger scale; this month 105 samples were examined, with the following results:—

	Colonies per c.c.
Thames water, unfiltered	6409
Thames water, from the clear water wells of the five Thames-derived supplies.. highest	75
Ditto ditto lowest	4
Ditto ditto .. (45 samples) mean	30
New River water, unfiltered	1420
New River water, from the Company's clear water well	37
River Lea water, unfiltered	1460
River Lea water from the East London Com- pany's clear water well	20

With regard to the above results, it is our duty to say that one very exceptional result was also recorded during the month, viz., on the 14th, when one of the wells was found to contain, in our judgment, too great a number of microbes, having regard to the usual high quality of the filtered supply. A serious warning of the abnormal bacteriological condition of the water was immediately communicated to the Engineer of the Company, and the next sample of water, taken within 48 hours, had resumed its normal condition.

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

THIRD ANNUAL REPORT OF COMMITTEE ON ATOMIC WEIGHTS. RESULTS PUBLISHED DURING 1895.*

By F. W. CLARKE.

(Continued from p. 90).

Tellurium.—The determinations of atomic weight by Staudenmeier (*Ztschr. Anorg. Chem.*, x., 189; calculations based upon $O = 16$ and $H = 1.0032$) all start out from telluric acid, $H_2TeO_4 \cdot 2H_2O$, which had been purified by repeated crystallisation. Two essentially different methods were adopted. First, telluric acid was dehydrated, and reduced to TeO_2 by heating. Secondly, telluric acid was reduced by heating in hydrogen to metallic tellurium, finely divided silver being mixed with the acid to retain the tellurium by preventing volatilisation. In four experiments, TeO_2 was reduced to Te in

the same manner. The weights and results may be classified as follows for the convenience of comparison:—

$TeO_2 : Te$.

TeO_2 .	Loss on reduction.	Atomic weight of Te.
0.9171	0.1839	127.6
1.9721	0.3951	127.7
2.4115	0.4835	127.6
1.0172	0.2041	127.5

Telluric Acid : TeO_2 .

Telluric acid.	Loss.	Atomic weight of Te.
1.7218	0.5260	127.2
2.8402	0.8676	127.1
4.0998	1.2528	127.1
3.0916	0.9450	127.05
1.1138	0.3405	127.0
1.9843	1.5236	127.05
4.6716	1.4278	127.1

Telluric Acid : Te.

Telluric acid.	Loss.	Atomic weight of Te.
1.2299	0.5471	127.3
1.0175	0.4526	127.3
2.5946	1.1549	127.2

There is a good discussion in the paper as to the possible cause of error in these determinations, and also concerning the place of tellurium in the periodic system. Staudenmeier upholds the homogeneity of tellurium as an element, as against the supposition that it is a mixture.

Some years ago Brauner, in an elaborate paper upon tellurium, sought to show that the ordinary element was a mixture of true tellurium with a higher homologue of atomic weight 214. He now (*Fourn. Chem. Soc.*, lxvii., 549) concludes that this is very improbable, and suggests that tellurium may contain a homologue of argon, of atomic weight 130. For this supposition no evidence is given apart from the abnormality of the atomic weight, which should fall below that of iodine.

Yttrium.—The atomic weight of this metal has been re-determined by Jones (*Am. Chem. Journ.*, xvii., 154; calculations based upon $O = 16$ and $H = 1.0032$), who started out with material purified by Rowland's process; that is, by precipitation with potassium ferrocyanide. First, oxide was converted into sulphate; and secondly, sulphate was transformed to oxide by calcination. The weights and results were as follows:—

First Method.

Y_2O_3 .	$Y_2(SO_4)_3$.	Atomic weight of Y.
0.2415	0.4984	88.89
0.4112	0.8485	88.92
0.2238	0.4617	88.97
0.3334	0.6879	88.94
0.3408	0.7033	88.90
0.3418	0.7049	89.05
0.2810	0.5798	88.94
0.3718	0.7803	88.99
0.4379	0.9032	89.02
0.4798	0.9901	88.91

Mean.. .. 88.94

Second Method.

$Y_2(SO_4)_3$.	Y_2O_3 .	Atomic weight of Y.
0.5906	0.2862	88.91
0.4918	0.2383	88.89
0.5579	0.2705	89.03
0.6430	0.3117	88.99
0.6953	0.3369	88.89
1.4192	0.6880	88.99
0.8307	0.4027	88.99
0.7980	0.3869	89.02
0.8538	0.4139	88.99
1.1890	0.5763	88.96

Mean.. .. 88.97

* Read at the Cleveland Meeting, December 31, 1895. From the *Journal of the American Chemical Society*, xviii., No. 3.

These determinations are probably the best hitherto made, although they have been briefly criticised by Delafontaine (CHEM. NEWS, lxxi., 243), who prefers the lower value obtained by himself, $Y=87.3$. Delafontaine reaffirms the existence of phillipium, and regards gadolinium as identical with decipium. Jones (CHEM. NEWS, lxxi., 305), in a brief rejoinder, defends his own work, and urges that Delafontaine has failed to show wherein it is defective.

The Cerite Earths.—Papers upon this subject have been published by Schützenberger and by Brauner. In his first communication, Schützenberger (*Compt. Rend.*, cxx., 663) deals with cerium, which had been freed from lanthanum and "didymium" by fusion of the mixed nitrates with saltpetre. The yellowish white cerium oxide was converted into cerium sulphate, which was dried at 440° . In this salt, with special precautions, the sulphuric acid was estimated by precipitation with barium chloride. One hundred parts of cerium sulphate gave 123.30 of barium sulphate. Hence, $Ce=139.45$, according to Schützenberger's calculations. Re-computing with—

$$O=16, S=32.07, \text{ and } Ba=137.43, \\ Ce=139.96.$$

In a second paper (*Compt. Rend.*, cxx., 962) Schützenberger describes the results obtained by the fractionation of cerium sulphate. Preparations were thus secured giving oxides of various colours, such as canary-yellow, yellowish rose, reddish, and brownish red. These, by the synthesis of the sulphates, the barium sulphate method, &c., gave varying values for cerium, ranging from 135.7 up to 143.3. Schützenberger concludes that the cerium sesquioxide from cerite contains small quantities of another earth of lower atomic weight. In a third paper (*Compt. Rend.*, cxx., 1143) he continues the investigation with the other cerite earths. For the didymiums he finds a range in atomic weight from 137.5 to 143.5 approximately.

Brauner's paper (CHEM. NEWS, lxxi., 283) is partly a reclamation of priority over Schützenberger, and partly a preliminary statement of new results. In his earlier work he found that cerium oxide was a mixture of two earths; one white, the other flesh colour with a tinge of orange, and atomic weights for the contained metal of 140.2 and 145.72 respectively. In his later researches Brauner fractionates his material by several methods. One constituent obtained from cerium oxide is a dark salmon-coloured earth, the oxide of a metal which he calls "metacerium." The other constituent he calls cerium. Pure cerium oxalate by Gibbs's permanganate method gave 29.506 and 29.503 per cent of cerium sesquioxide with 46.934 per cent of cerium dioxide. Hence, $Ce=139.91$, or, with a slight correction, $Ce=140.01$. This is not far from Schützenberger's value.

(To be continued).

THE DETERMINATION OF ATOMIC MASSES OF SILVER, MERCURY, AND CADMIUM, BY THE ELECTROLYTIC METHOD.*

By WILLETT LEPLEY HARDIN.

(Concluded from p. 93).

PART III. (continued).

FIRST SERIES.

Experiments on Cadmium Chloride.

DUMAS and Bucher have both determined the ratio of cadmium to chlorine in cadmium chloride. The results given for the atomic mass of cadmium by the latter ex-

perimenter are almost four-tenths of a unit higher than those given by the former.

Preparation of Cadmium Chloride.

Hydrochloric acid was purified by first passing chlorine through the commercial C. P. acid to remove any sulphur dioxide; the excess of chlorine was removed by a current of carbon dioxide. The acid was then distilled from calcium chloride and the hydrochloric acid gas collected in pure water. Pure metallic cadmium was then dissolved in the acid and the solution evaporated to crystallisation. The crystals of cadmium chloride were removed from the liquid and thoroughly dried. The material was then placed in a hard glass combustion-tube, similar to that used in the distillation of metallic cadmium, and carefully sublimed in a current of dry carbon dioxide. The first and last portions of the sublimate were rejected. The middle portion, which consisted of pearly leaflets, was placed in a weighing tube and kept in a desiccator. As only a small quantity of the material could be sublimed at a time, the different analyses were made from different sublimations.

Mode of Procedure.

A weighed portion of the cadmium chloride was dissolved in a little water in a platinum dish, a slight excess of potassium cyanide was added and, after diluting to 200 c.c. with pure water, the solution was electrolysed. Before interrupting the current, the liquid was syphoned from a dish in a manner already outlined in the experiments on silver. The metallic deposit was washed several times with boiling water and carefully dried. The strength of the current and time of action were as follows:—

Time of action.	Strength of current.
12 hours ..	N.D ₁₀₀ =0.1 ampère.
4 " ..	N.D ₁₀₀ =0.15 "
4 " ..	N.D ₁₀₀ =0.30 "

The cadmium was thrown down as a dense white deposit. Ten results on cadmium chloride reduced to a vacuum standard on the basis of—

3.3 =	density of cadmium chloride,
8.55 =	" metallic cadmium,
21.4 =	" platinum dish,
8.5 =	" weights,

and computed for the formula $CdCl_2$, assuming 35.45 to be the atomic mass of chlorine, are as follows:—

	Weight of $CdCl_2$. Grms.	Weight of Cd. Grm.	Atomic mass of cadmium.
1	0.43140	0.26422	112.054
2	0.49165	0.30112	112.052
3	0.71752	0.43942	112.028
4	0.72188	0.44208	112.021
5	0.77264	0.47319	112.036
6	0.81224	0.49742	112.023
7	0.90022	0.55135	112.041
8	1.02072	0.62505	112.002
9	1.26322	0.77365	112.041
10	1.52344	0.93314	112.078

Mean = 112.038
Maximum .. = 112.078
Minimum .. = 112.002

Difference .. = 0.076
Probable error = ± 0.005

From the total quantity of material used and metal obtained, we have 112.040 for the atomic mass of cadmium.

SECOND SERIES.

Preparation of Cadmium Bromide.

The bromine used in this series was purified as outlined in the experiments on mercuric bromide. The

* Contribution from the John Harrison Laboratory of Chemistry, No. 13. From the author's thesis presented to the Faculty of the University of Pennsylvania for the degree of Ph.D.—From the *Journal of the American Chemical Society*, xviii., p. 990.

cadmium bromide was prepared by allowing bromine water to act on metallic cadmium for several days at the ordinary temperature. When the action was complete, the solution was filtered and evaporated to crystallisation. The crystals of cadmium bromide were removed from the liquid and thoroughly dried. The material was then placed in a hard glass combustion-tube and carefully sublimed in a current of dry carbon dioxide. The first and last portions of the sublimate were rejected. The middle portion was removed from the tube, placed in a weighing bottle and kept in a desiccator. The product obtained in this way consisted of a crystalline pearly leaflet which dissolved immediately in water without leaving a residue.

Mode of Procedure.

The method of operation was the same as for cadmium chloride. A weighed portion of the material was dissolved in a little water in a platinum dish. A slight excess of potassium cyanide was then added, and after diluting to 200 c.c. the solution was electrolysed and the resulting metal weighed. The strength of current and time of action were the same as for cadmium chloride.

Ten observations on cadmium bromide reduced to a vacuum standard on a basis of—

4.8	= density of cadmium bromide,
8.55	= " metallic cadmium,
21.4	= " platinum dish,
8.5	= " weights,

and computed for the formula CdBr_2 , assuming 79.95 to be the atomic mass of bromine, are as follows:—

	Weight of CdBr_2 . Grm.	Weight of Cd. Grm.	Atomic mass of cadmium.
1	0.57745	0.23790	112.031
2	0.76412	0.31484	112.052
3	0.91835	0.37842	112.067
4	1.01460	0.41808	112.068
5	1.15074	0.47414	112.053
6	1.24751	0.51392	112.019
7	1.25951	0.51905	112.087
8	1.51805	0.62556	112.076
9	1.63543	0.67378	112.034
10	2.15342	0.88722	112.041
Mean = 112.053			
Maximum .. = 112.087			
Minimum .. = 112.019			
Difference .. = 0.068			
Probable error = ± 0.005			

From the total quantity of material used and the metal obtained, $\text{Cd} = 112.053$.

THIRD SERIES.

In these experiments an attempt was made to determine the ratio of the atomic mass of cadmium to that of silver by allowing the same electric current to pass successively through solutions of the two metals and weighing the resulting deposits. The arrangement of apparatus and the details of the method were described under the mercury-silver series. The results were not as satisfactory as the corresponding results obtained for mercury. A large number of determinations were made with currents of different strength and solutions of different concentration, but the results were, in most cases, far below those obtained in the first two series. A current which deposited about twelve-hundredths of a gram. of silver per hour seemed to give the best results. From all the observations, five results were selected which differed only about one-tenth of a unit from those of the first two series. Results selected in this way are entitled to but little weight, and perhaps should not be used in determining the general mean of all the observations.

Computed on the basis of 107.92 for the atomic mass of silver, the only admissible results are as follows:—

	Weight of Ag. Grm.	Weight of Cd. Grm.	Atomic mass of Cadmium.
1	0.24335	0.12624	111.928
2	0.21262	0.11032	111.991
3	0.24515	0.12720	111.952
4	0.24331	0.12616	111.916
5	0.42520	0.22058	111.971

Mean = 111.952
Maximum .. = 111.991
Minimum .. = 111.916

Difference .. = 0.075

This method was discussed under mercury. The probable sources of error pointed out there apply equally well in the case of cadmium. Until the large variations can be accounted for and the difficulties overcome, the method must be regarded as unsatisfactory.

Summary.

Inasmuch as but one method of analysis has been used throughout this work, it is useless to discuss it here. The advantages and objections pointed out under silver apply also to cadmium.

In summing up the work on cadmium, equal weight must be given to the first two series. The last series must be considered alone, and all that need be said of it is, that the results obtained for the atomic mass of cadmium never exceeded 112. In the corresponding series on mercury, the variations were in both directions from 200.

The general mean of the first two series calculated from the separate observations is—

Atomic mass of Cd.		
First series	= 112.038	
Second series	= 112.053	
General mean	= 112.0455	

From the total quantity of material used and metal obtained we have—

Atomic mass of Cd.		
First series	= 112.040	
Second series	= 112.053	
General mean	= 112.0465	

Combining this with the first general mean we have 112.046 as the most probable result of all the work, for the atomic mass of cadmium. This result is lower than those obtained by Huntington and Bucher, but agrees very closely with the results obtained by von Hauer, Dumas, Lensen, Jones, and Lorimer and Smith.

I wish here to express my sense of gratitude to Professor Edgar F. Smith, at whose suggestion this work was undertaken and under whose personal supervision it was carried out.

Radio-photography of the Soft Parts of Man and of the Lower Animals.—MM. Remy and Contremoulin. We have the honour of presenting to the Academy a new result of our researches on the application of the X rays to anatomical studies. By the aid of chemical preparations on the bodies of men and frogs, we have been able to place the muscles, the ligaments, and the tendons in such a state that they have yielded radio-photographic images. The muscle projected shows a dark tint, but within the limits thus indicated we perceive dark traits which pertain to the muscular bundles. The muscle is thus masked by bundles of longitudinal striae very distinctly limited. In the frog, prepared by the same means, the muscles are fully visible. In this animal we have obtained an image of the crystalline lens and of the coatings of the eye.—*Comptes Rendus*, cxxiv., No. 5.

ELECTRIC SHADOWS AND LUMINESCENCE.*

By Prof. SILVANUS P. THOMPSON, D.Sc., F.R.S., M.R.I.

The early days of the year 1896 were marked by the announcement telegraphed from Vienna to the effect that Professor Röntgen, a man whose name, though little known outside the world of science, was well known and highly esteemed by those who were initiates in physics, had discovered the existence of rays of a new and extraordinary kind. Taking a Crookes tube, excited of course by a proper electric spark, and covering it up within a case of black cardboard, he found it to produce in the surrounding space some entirely unexpected effects. Black cardboard is impervious not only to ordinary light and to radiant heat, but also to all those other known kinds of invisible light beyond the violet end of the spectrum, known as actinic waves, which are such active agents in the production both of fluorescence and of photographic actions. Yet the invisible emanations of the Crookes tube, which passed freely through the opaque cardboard, were found by Röntgen to be capable of revealing their presence in two ways. In the first place he had seen them to project shadows upon a luminescent screen of paper coated with the highly fluorescent substance called platino-cyanide of barium, and in the second place he had been able to photograph these shadows by letting them fall upon an ordinary photographic plate. The discovery was singular. It revealed the existence of a remarkable and hitherto unexpected species of radiation. It added another to the many puzzling phenomena attendant upon the discharge of electricity *in vacuo*. It proved that something which in the ordinary sense in which those terms are used is neither light nor electricity was generated in the Crookes tube, and passed from it through substances opaque alike to both.

But that which took the imagination of the multitude by storm, and aroused an interest the intensity the like of which has not been known to be aroused by any other scientific discovery in our times, was not the fact that Professor Röntgen had seen luminescent shadows from a Crookes tube, or had obtained a photograph of those shadows; it was the entirely subsidiary and comparatively unimportant point that to these mysterious radiations flesh is more transparent than bone.

Let me begin by showing you as a first experiment that same fact which Röntgen announced of the production of luminescent shadows by these invisible rays. Before you there stands a Crookes tube, of the most modern kind,† for this particular purpose. We have here an induction coil‡ capable of giving 6-inch sparks, with which we can send electric discharges through the tube, illuminating it with its characteristic golden-green glow. I now cover over the tube and exclude all ordinary light, not with a box of black cardboard but with a black velvet cloth. And now in the darkness I am able to show you how on a sheet of paper covered with the highly fluorescent platino-cyanide of barium—the well-known substance which Röntgen himself was using—the shadows of objects placed between. See how this sheet shines in the light of the tube transmuting the invisible radiations into visible light. I hold my purse behind the screen—you see the shadow of the metal clasp, and of the metal contents (two coins and a ring), but you see not the shadow of the leather purse itself, for leather is transparent to these rays while metal is opaque. I hold my hand behind and you see—or at least those of you who are within a few yards of me—the shadow of my hand, or rather of the bones of my hand, surrounded by a fainter shadow of the almost transparent flesh.

* A Lecture delivered at the Royal Institution of Great Britain Friday, May 8, 1896.

† A Crookes "focus" tube (Jackson pattern), constructed by Messrs. Newton and Co., of Fleet Street, London.

‡ An Appa coil capable of giving sparks 25 centimetres in length, but on this occasion excited with only 5 cells, giving sparks about 6 inches in length.

Now the second fact that Röntgen announced was that these same rays which escape through the opaque covering and excite fluorescence are also capable of taking photographic impressions of the shadows. There is nothing whatever new about this part of the subject: it is the old photograph; there is no "new photography." Here is a common camera back, and here inside it is a photographic dry-plate—quite a common dry-plate, such as has been known for ten years. This plate is covered with a black card, so that it may not become fogged by the light of the room when I draw the slide. All I have to do is to lay it upon the table below the Crookes tube so as to cast the shadow upon it, and after due exposure develop the plate in the ordinary well-understood way. Now it may be interesting to see the proof of the fact that bone is less transparent than flesh. So, with your permission, I will ask my little daughter to have her hand photographed. (Experiment made.)

At the time of Röntgen's announcement, the exposure required with the Crookes tubes that were then in existence was from twenty minutes to, I think, two or three hours. Very shortly improvements were made; and with these modern tubes one minute is quite sufficient for an exposure. Indeed, one minute is too much for many objects. I have not previously tried this particular tube, though I judge by its appearance that it is in good condition. As soon as the exposure of one minute is over we will have the plate taken into the dark room and developed in the ordinary way; and when it is developed we will have it brought back into this room and put into the lantern, that you may see what has been done.

Now, while we are taking photographs, I may as well take a second to illustrate another point. Röntgen investigated in the most careful and elaborate way the relative transparency of different materials for these mysterious rays. He noticed that wood, and many substances which are opaque to ordinary light, are transparent to these rays; whilst, on the contrary, several substances that are transparent to light, such as calc-spar and heavy glass, are very opaque toward them. Many experimenters have examined this question of relative transparency. I devoted a day or two to the study of gems, and found that imitation rubies made of red glass are much more opaque than real rubies, and that paste diamonds are much more opaque than real diamonds. Real diamonds and rubies are indeed very transparent, and scarcely cast any shadows on the luminescent screen, though I have found diamond to be more opaque than an equal thickness of black carbon. There are laid upon this piece of card two rubies, one being only a glass ruby. There is also a row of four small diamonds. I will leave you to find out whether they are false or real. And then there are three larger diamonds, one of which is uncut and is a genuine South African stone. I lay them down upon a photographic plate and expose them to the Röntgen rays so that we may test their relative transparency. (The two photographs thus taken were projected upon the screen at the close of the lecture.)

Amongst the things which Röntgen told us was the fact that different kinds of glass are unequally transparent; that lead-glass, for instance, is much more opaque than soda-glass, or potash-glass, or, indeed, any glass which does not contain a heavy metal like lead. He found that practically the transparency was governed by the density; that the heavy or the dense substances were the more opaque. There is now some reason to correct that statement, though in the main as a first approximation it is perfectly true. Professor Dewar has shown that you must take into account, not the density in gross, but the atomic weight. Taking any homologous series, for example, such as a number of sulphides, or oxides, or chlorides, that one which contains the atomically heavier metal will be the more opaque. Again, the bromide of sodium is more opaque than the chloride of the same metal, and the iodide is more opaque than the bromide. But as the correspondence between relative

opacity and molecular or atomic weight breaks down when we try to pass from one series of compounds to a different series, there is some reason to carry the matter to a further degree of approximation. We must go beyond the suggestion of atomic weight. The nearest approach to a law that I have been able to get at yet, on comparing tables of statistics, is that the transparency is proportional to the specific heat. For homologous series this is, of course, the same as saying that the transparency is inversely proportion to the molecular weight.

Röntgen found all the heavy metals to be remarkably opaque, while light metals like sodium and aluminium, and even zinc, are remarkable for their transparency. Aluminium, which is opaque to every known kind of light, is transparent, even in sheets half an inch thick, to these rays. Lithium, the lightest of solid metals, and with an atomic weight 7 as against aluminium 27, is so transparent that I have not been able yet even to see its shadow. Of all liquids water is the most transparent, and it has the highest specific heat of all of them.

Röntgen further found these rays to be incapable either of refraction by lens or prism,* or of reflection by any polished mirror. Reflection there is in one sense, that of diffuse reflection, such as white paper exercises on common light. No lens can concentrate these rays; they are also apparently incapable of being polarised. One difficulty in experimenting on these strange properties is that air itself acts as a turbid medium, reflecting back diffusely, and a smoky cloud would do for ordinary light, a portion of the rays.

Finding that these radiations differed in so many ways from ordinary light, and while resembling and even surpassing ultra-violet rays in their strong actinic properties, yet differed entirely from them in respect of the properties of refraction, reflection, and polarisation, he named them "X rays." To judge by his own writing, he appeared to wish that they might prove to be longitudinal vibrations in the ether, the possibility of the existence of which has been a subject of speculation on the part of some of the most learned of mathematical physicists. Others have suggested that these X rays are transverse vibrations of a much higher frequency and shorter wave-length than any known kind of ultra-violet light. Others, again, see in them evidence that radiant matter (*i.e.*, cathodic streams of particles) can traverse the glass of a Crookes tube, and regard them as material in their nature. Lastly, it has been suggested that they may be neither waves nor streams of matter, but vortex motions in the ether.

To follow out the bearings of these speculations, as well as to trace the development of discovery, let us go back a little and consider what was the starting-point of Röntgen's research. He was using a Crookes tube. It is one of the difficulties of my task to-night that I have to speak in the presence of him who is the master of us all in this subject of electric discharges in the vacuum tube. But to understand the discoveries of Crookes let us first witness a few experimental illustrations of the phenomena of electric discharges in vacuum tubes. Many of them have been known for half a century. We all know of the researches made in England by Gassiot, and by Varley and others, and the tubes of Geissler of Bonn are a household word. But there is one set of researches which deserves to be known far better than it is, that made by Dr. W. H. Th. Meyer, of Frankfort, whose pamphlet† I hold in my hand. In it he depicts a number of tubes in various stages of exhaustion, including one in

that highest stage of exhaustion which one is prone to think of modern origin.

In order to illustrate the successive phenomena which are produced when electric discharges are sent through a tube during progressively increasing exhaustion, there is here exhibited a set of identical tubes. Each is a simple straight tube, having sealed in at each end an electrode terminating in a short piece of aluminium wire. The electrode by which the electric current enters is known as the anode, that by which it leaves the tube as the cathode. The only difference between these eight tubes lies in the degree of rarefaction of the interior air. The first one contains air at the ordinary pressure. As its electrodes are about 12 inches apart I am unable with the Apps induction coil (excited to throw an 8-inch spark) to send a spark through it. From the second tube about four-fifths of the air has been abstracted, and here we obtain a forked brush-like spark between the electrodes. The third tube has been exhausted to about one-twentieth part, and shows as the discharge a single thin red linear spark like a flexible luminous thread. When, as in the fourth tube, the exhaustion is carried so far as to leave but one-fortieth, the red line is found to have widened out into a luminous band which extends from pole to pole, while a violet mantle makes its appearance at each end and spreads over both of the electrodes. On carrying the exhaustion to the stage shown by the fifth tube, where only about 1-500th of the original air is left behind, we note that the luminous column has broken up transversely into flickering striæ, that the violet mantle round the cathode has become more distinct, and is separated by a dark interval from the luminous red column, while a second and very narrow dark space appears to separate the violet mantle from the surface of the cathode. In the sixth tube the exhaustion has been carried to about 1-10,000th. The flickering striæ have changed shape and colour, being paler. The light at the anode has dwindled to a small bright patch. The violet glow surrounding the cathode has expanded to fill the whole of that end of the tube; the dark space has become more distinct, and within it the cathode now shows on its surface an inner mantle of dull red light. There is a slight tendency for the glass to show a greenish fluorescence near the cathode end. In the seventh tube the luminous column has subsided into a few greyish white nebulous patches, the dark space round the cathode has greatly expanded, and the glass of the tube has now begun to show a yellow-green fluorescence. The exhaustion has been pushed so that only about 1-50,000th or less of the original air is present. In the eighth and last tube only one or two millionths of the original air have been left, with the result that the tube now offers an enormously increased resistance to the passage of the discharge. All the internal flickering nebulosities have vanished; the tube looks as though there were no residual air within. But now the glass itself shines with a fine yellow-green fluorescence which is particularly bright in the region around the cathode. Were the exhaustion to be carried much further the spark from this induction coil would no longer pass, so high would the resistance become. All these successive stages up to the last can be shown in one and the same tube attached to a modern rapid air-pump. But for the proper production of the high vacua of the last stages, where electric shadows are alone produced, nothing short of a mercurial pump, either in the form invented by Sprengel or in that used by Geissler (or one of the recent modifications) will suffice.

The phenomenon of fluorescence of the glass, which manifests itself when the exhaustion has become sufficiently high, was known in a general way as far back as 1869 or 1870. The tube next to be shown is a modern reproduction of a tube used at that time by Hittorf, of Münster. It differs from the tubes last shown by having a bend in it. Hittorf observed that when such a tube is exhausted sufficiently highly to give at the cathode the characteristic greenish yellow fluorescence, this greenish

* Perrin, in Paris, and Winkelmann, in Jena, have independently found what they believe to be evidence of refraction through an aluminium prism. Both observers detected a slight deviation, but in a direction toward the refracting angle, showing aluminium to have for these rays a refractive index, slightly less with respect to air than unity.

† "Beobachtungen über das geschichtete elektrische Licht, sowie über den merkwürdigen Einfluss des Magneten auf dasselbe;" von Dr. W. H. Theodor Meyer. Berlin, 1898.

yellow fluorescence refused to go round the bend. It might appear at one end or the other, according to the direction in which the discharge was being sent, but would not go round the bend. The effect was as if the discharge went in straight lines from the bit of wire that served as cathode to the walls of the tube. Indeed shadow effects were observed by him, and by Wright, of Yale, and afterwards independently by Crookes, who greatly extended our knowledge of the facts. We may take this fact, that the fluorescence caused by the kathode will not go round a corner, as the starting-point of the memorable researches of Crookes on radiant matter a score of years ago.

Before you are several tubes which illustrate the researches made by Crookes. The first is a simple glass bulb into which are sealed the two electrodes,—the anode, by which the current enters, terminating in a bit of stout aluminium wire; the other, by which the current leaves, called the cathode, terminating in a small flat aluminium disk. The glass bulb was itself highly exhausted—how highly we shall presently see. From the flat front surface of the cathode, when sparks are sent through the bulb, a sort of back-discharge takes place in a direction normal to the surface. This discharge, which only occurs at a very high degree of exhaustion, possesses several properties which distinguish it from all other kinds of discharge. It is propagated in straight lines, causes a brilliant luminescence wherever it strikes against the glass walls of the tubes, casting shadows of intervening objects, it heats the surface on which it impinges, and strikes them with a distinct mechanical force. Singular to relate, it is also capable of being deflected by a magnet as though it were a flexible conductor carrying the current. Struck by the singularity of these kathode rays or kathode discharges, which formed the subject of several beautiful researches,

in fact, the direction of these kathode rays was found to be independent of the position of the anode. He found kathode rays to be produced even when no internal electrodes were inserted, and when, instead, external patches of tinfoil were attached to the glass. Their mechanical action he studied by causing them to impinge upon the vanes of a pivoted fly, which was thereby set into rotation. In a later experiment he caused the fly of a "molecule mill" to be set into rotation, not by the impact of the kathodic discharge, but by the kinetic energy of the particles returning back toward the anode after they had impinged against the walls of the tube and lost their negative electric charges. A mere *resumé* of Crookes's work in those years beginning about 1869 or 1870, and extending not only for ten years actively, but going on at intervals until a year or two ago, would of itself fill a whole course of lectures. Into the controversy which has arisen between Crookes and the English physicists on the one hand, and the German physicists on the other, there is no need to enter. Suffice it to say that while the German physicists mostly reject Crookes's hypothesis of radiant matter, and regard all these various phenomena as the result of mere wave-motions within the tube, the British physicists, including Lord Kelvin and Sir George Stokes, accept Crookes's view of the material nature of the kathode rays. Who, indeed, that has seen the molecule mill at work can doubt that, whether vibrations are present or not (and doubtless there are vibrations present), there are actually streams of moving particles as an essential feature of the kathodic discharge? For the moment the victory undoubtedly rests with the views of Crookes.

But of all these phenomena the one which concerns us most is that of the production of electrical shadows. Erecting in the path of the kathode rays an obstacle cut

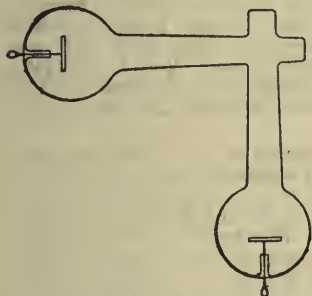


FIG. 1.

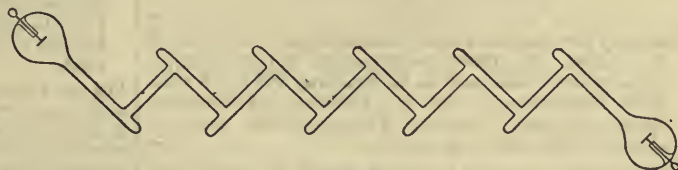


FIG. 2.

Crookes advanced the hypothesis that they consisted of flights of negatively electrified particles or "radiant matter." The particles he sometimes spoke of as molecules, sometimes as dissociated atoms, or, as we should now say, ions. He studied the wanderings of these flying particles by inserting within the bulb at different points auxiliary electrodes. He found the interior of the bulb to be positively electrified in all parts except within the dark space which surrounds the cathode, that is to say, except within the range of the actual kathode discharge. The kathode discharge itself was found to be possessed, to an extent exceeding any other known agency, of the power of exciting fluorescence and phosphorescence in minerals and gems. The kathode rays were themselves discernible as they crossed the interior of the tube. In such a bulb the kathode rays would form a blue streak impinging straight upon the anode. The kathode used in the next Crookes tube is of a concave shape. Crookes found that, since the kathode rays left the surface normally, the result of curving the kathode was to focus the rays toward the centre of curvature. By so focussing the rays upon a bit of platinum foil, it was found possible to fuse and even melt the metal.

Unlike the discharges obtained at lower stages of rare-

out in sheet metal,—a cross of thin aluminium is the favourite object,—a shadow of it is observed to be cast upon the wall of the tube behind it; the glass phosphorescing brilliantly except where shielded from the impact of the kathode rays, so that the shadow comes out dark against a luminous background. Common soda-glass gives this greenish golden tint, while lead-glass exhibits a blue phosphorescence. Not glass alone, but diamonds, rubies, emeralds, calc-spar, and other earthy materials, such as alumina, and notably yttria, produce the most brilliant effects under the kathode discharge, some of them only fluorescing transiently, others with a persistent phosphorescence. As a sample is shown a tube in which a sea-shell, slightly calcined to remove organic matter, is made to emit a brilliant luminescence under the impact of rays from a kathode placed above it. The shell itself casts a shadow against the lower part of the tube. Some of the shadow effects are very mysterious, and have recently claimed much of my attention. The size of the kathodic shadows is affected by the electrical state of the object. Electrifying it positively makes its shadow shrink to smaller dimensions. Electrifying it negatively causes a singular enlargement of the shadow. There seems to be no difference between the shadow of a metallic body

and that of a non-metallic body of the same size. All bodies cast shadows, however thin. Even a film of glass 1-10,000th of an inch thick—so thin that it showed iridescence like a soap-bubble—was found by Crookes to cast its shadow.

Another point noticed by Crookes was that if the exhaustion is carried very far, and the tube is stimulated by a sufficiently strong electromotive force, the phosphorescence may occur at points not in the line of discharge, but round a corner. Not that the kathode rays turn the corner, however. Apparently some of the more quickly moving, or perhaps more highly charged particles,—atoms, molecules, or ions, those, in fact, described by Crookes as "loose and erratic,"—would manage to get round the corners and produce effects of a more or less directly cathodic kind in places where they could not have penetrated by any motion in a straight line.

Here (Fig. 1) is a tube—a variation on one of Hittorff's. having two branches that cross one another at right angles. There are two small disks of aluminium in the bulbous ends to serve as electrodes. When either of these is made the kathode, the whole limb in which it is situated fluoresces brilliantly of a golden-green tint, particularly at the distant end. But the other limb remains dark, save for a little nebulous blue patch, near the anode, due to residual gas. Another tube (Fig. 2) is made as a zigzag, and here again only the end branch shines. On reversing the current the luminescence shifts to the other end. But when the tube is more highly exhausted, the phosphorescence is observed not only in the end branch where the kathode is, but also slightly at the end wall of each branch of the zigzag. Apparently the residual gas will act partly as its own kathode, and throw off something which causes the glass beyond to phosphoresce.

(To be continued).

NOTICES OF BOOKS.

Chemistry for Engineers and Manufacturers. A Practical Text-book. By BERTRAM BLOUNT, F.I.C., F.C.S., Assoc. Inst. C.E., and A. G. BLOXAM, F.I.C., F.C.S. With Illustrations. Vol. II., *Chemistry of Manufacturing Processes.* London: Charles Griffin and Co. (Ltd.). 1896. 8vo., pp. 484.

THIS volume is devoted to notices of the sulphuric acid manufacture; of alkali and its by-products; of destructive distillation, including the gas-manufacture; of artificial manures, petroleum, lime, and cement; of the clay industries, and glass, sugar, and starch; of brewing and distilling; oils, resins, and varnishes, soaps and candles, textiles and bleaching colouring-matters, dyeing and printing; of paper and pasteboard, pigments and paints, leather glue and size, explosives and matches; and of the minor chemical manufactures.

The reader will be surprised at finding all these arts and manufactures—some of capital importance—treated in the compass of 437 pages; but the authors explain in their Preface that they seek to expound those dominant principles which, they allege, "are too often hidden beneath masses of mere detail, and are consequently apt to be overlooked by the specialist in any one branch, to his detriment, in that he frequently fails to apply to his own work principles which are matters of common knowledge elsewhere."

It is to be regretted that no instances are given of this overlooking which so frequently happens. According to our own observation the specialist eagerly, and even anxiously, looks about for principles which may throw light upon his own department.

The bibliography seems to us somewhat deficient; not a few important works on different departments have been overlooked.

We regret to find that the authors have omitted the opportunity to deal a blow in passing at the recent stultification of the Methylated Spirit A.G. If the Excise wished to render methylated spirit absolutely undrinkable, they might have demanded the addition to the spirit of a few drops of Dippel's animal oil, which is successfully used in Germany, and which does not interfere with industrial uses.

The addition of dyes and mordants to sugar—an increasing evil—should be carefully looked into.

Mention is made of a fraudulent custom in the pigment-trade, pale chrome-yellows being called and sold as pure when let down with lead sulphate. Hard waters for the use of the dyer and tissue-printer are rightly objected to; if a hard water is required for any special process, it is better to add a salt of lime to a pure-water supply.

The manufacture of artificial silk is considered as hitherto not a commercial success.

In the matter of the vinegar manufacture, it may be asked why sugar at its present prices is not used in preference to malt, save for domestic manufacture? No mention is made here of date vinegar, which is coming into use.

This work will be found useful to manufacturers who, without being chemical specialists, wish to have a general insight into chemical manufactures.

A Practical Treatise on Animal and Vegetable Fats and Oils, both Fixed and Volatile; their Physical and Chemical Properties and Uses, the Manner of Extracting and Refining them, and Practical Rules for Testing them—as the Manufacture of Artificial Butter, of Lubricants, &c. With Lists of American Patents relating to the Extraction, Rendering, Refining, Decomposing, and Bleaching of Fats and Oils. By WILLIAM T. BRANNT, Editor of the "Techno-chemical Receipt-Book," "Petroleum," &c. Second Edition, Revised and in great part Re-written. Illustrated with 302 Engravings. In Two Volumes. Vol. I., pp. 528; Vol. II., pp. 728. Philadelphia: H. C. Baird and Co. London: Sampson Low, Marston, and Co. (Ltd.). 1896.

WE have here an encyclopædia of the animal and vegetable fats and oils from a botanical, chemical, industrial, and commercial point of view. The analysis of oils, whether for identification or for the detection of frauds, presents great difficulties. Numerous processes, physical and chemical, have been applied, not without success. But the properties of oils are apt to be modified by age, by climate, by the soils of their native countries, so that, especially in forensic cases, it becomes very difficult for the expert to give an apodictic decision as to the genuine or fraudulent character of any sample in question. We have here a most elaborate table of the colour reaction, with a mixture of sulphuric and nitric acids, with potash and soda lye, with zinc chloride, with hydrochloric acid and sugar. Next come the well-known elaidin test, the thermal test of Maumené, and Fehling test, depending on the heat liberated on mixing fatty oils with sulphuric acid at density 1.840; Tomlinson's cohesion figures, which require the outlay of much time before trustworthy results can be reached.

Among quantitative methods there rank the determination of the ester number, of the acid number, the acid number + the ester number being the Kottstorfer saponification number; the determination of the fatty acids insoluble in water (Hehner's number); determination of the volatile fatty acids (Reichert's number); the iodine absorption (Hübl's number). The determination of oxy-fatty acids gives the acetyl number of Benedikt-Ulzer.

In determining the purity of an oil, the author examines firstly the argonoleptic and generally the physical properties. He then applies the qualitative chemical methods, and, lastly, the quantitative procedures,

among which Hübl's iodine number is most generally determined.

A table shows the approximate relative commercial value, and hence gives a key to the oils likely to be used for fraudulent purposes.

No small trouble is occasioned in commerce by the circumstance that the seeds of the three species of *Brassica*, which yield respectively colza, rape, and rubsen oils, cannot be decisively distinguished from each other, either by measurement or by the aid of the microscope.

The presence of any oil of this group—the products of the *Cruciferae*—may be detected by boiling the oil with white-lead plaster (*Emplastrum plumbi*). The oil is turned brown or black by the formation of lead sulphide.

Sesame oil or gingelly oil is used as a table oil, and is esteemed fully equal to the best olive oils. It is a curious fact that, though the seed is chiefly grown in India, yet the oils pressed in Europe are considered superior to those pressed at home.

Olive oil, in consequence of its high price, is especially open to fraud. Its electric conductivity is much lower than that of any other vegetable oil. Accordingly Palmieri has devised an apparatus—the *diazometer*—which utilises this peculiarity. This instrument, however, is expensive, and not easy of manipulation. Moreover, leaving sophistication out of the question, olive oils are subject to spontaneous alterations which render the test untrustworthy.

The spectroscopic examination of olive oil is likewise not trustworthy. The absorption bands observed in olive oil are not due to the oil itself, but to chlorophyll, and do not occur at all in bleached oils.

M. Brullé, Director of the Agricultural Station at Nice, has devised two methods of testing the purity of olive oils, showing the kind and quantity of other oils used for their sophistication.

These two tests we shall give *in extenso* on a future occasion. We must remember that dishonest merchants and manufacturers now consult experts who are themselves unscrupulous, or who are carefully kept in ignorance of the purposes for which their advice is required.

We shall return to this most valuable work at the earliest opportunity.

CORRESPONDENCE.

HOW SOON SHALL THE STUDENT BEGIN THE STUDY OF QUALITATIVE ANALYSIS?

To the Editor of the Chemical News.

SIR,—Mr. Beebe's article in your last issue is an interesting one, and will I hope bring some comment. I should think, however, that few teachers would agree with him, for surely qualitative analysis is an application of the science and not the science itself. The great difficulty is to induce pupils to *think*, and though one may be careful to point out that qualitative analysis is really a chemical Euclid, I have found but few pupils who will work through their analyses according to the syllogistic method of the great geometer. The attention given to schemes of analysis, and the importance given to the testing of powders until recently by the Science and Art Department have done much to take away the educational value of chemistry as a school subject.

As regards danger in making the common gases, these will have to be made some time, and if it is pointed out to a pupil why there is danger, and how to guard against it, no mishap is likely to occur, even should that "dangerous gas" hydrogen be the subject of the lesson.—I am, &c.,

C. J. WOODWARD.

Municipal Technical Schools,
Birmingham, Feb. 20th, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 4, January 25, 1897.

A medal was presented to M. Faye on the occasion of the 50th anniversary of his nomination as a Member of the Academy of Sciences. M. Faye made a suitable reply.

Fluorescence of Vitrified Matters under the Action of Röntgen's Rays.—M. [Ragiquet. — The following phenomena have, I believe, not been hitherto signalled. The substances mentioned below become luminous under the influence of the X rays, in the following decreasing order:—Baked enamels; crown glass; flint-glass; ordinary glass, and especially the kind known as crystal; sheet-glass from the works of Saint-Gobain; porcelain enamelled faience; enamel powder before baking; and even cut diamond. We know, also, that most of these substances are fluorescent in the violet and the ultra-violet rays. It is therefore possible to form with these substances fluorescent screens which enable us to repeat radiosopic experiments with this advantage, that the vitrified substances just mentioned may be worked optically. The images obtained are more definite, though less brilliant, than are those with crystals cemented upon card hitherto employed. We utilise also successfully these substances for shortening the exposure in radiographic experiments, and we have not to fear the granular spots produced by the crystals above mentioned. Does this fluorescence of glass not explain the disputed fact that persons affected with cataract see the X rays? In fact, if we place ourselves in the field of emission of a Crookes tube, furnished with thick spectacles with convex glasses, we experience the sensation of a light like that of phosphorus. This sensation is the result of the fluorescence of the glass, which forms before the eyes a luminous mist easily recognised by persons surrounding the patient. Besides the scientific applications there are an entire series of very beautiful experiments, which I am about classifying with a view to early publication.

Action of Carbon Dioxide and Monoxide upon Aluminium.—MM. Guntz and Masson. — This paper will be inserted in full.

Spectra of the Non-metals in Fused Salts—Silicon.—A. de Gramont. — This paper will be inserted in full.

On Chromium and Manganese Phosphides.—A. Granger.—Already inserted.

Influence of Temperature on Rotatory Power.—Ph. A. Guye and Mile. E. Aston.—In all we know at least fifty active liquids whose rotatory power decreases with the elevation of temperature in the entire interval of the experiments.

On Two Isomeric Triethylene-diphenyl Hydrazines, α and β .—In presence of hyposulphites the action of aldehyd or phenylhydrazine yields the isomer α almost pure. In neutral solutions aldehyd acting upon phenylhydrazine phosphate produces chiefly the isomer β .

On a High Homologue of Urea.—Oechsner de Koninck.—It seems very admissible that in proportion as the oxidising power of the system is weakened, the number of the atoms of carbon of the quaternary compounds eliminated by the kidneys increases progressively.

Contribution to the Study of the Action of Zinc upon Red Wines.—L. A. Lovat.—Zinc denaturates red wines and renders them poisonous. Hence the use of this metal should be severely forbidden in the cock for casks, vats, &c.—*Comptes Rendus*, cxxiv., No. 5.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Waterproofing Canvas.—I should like to know a good receipt for chemically rendering canvas, &c., waterproof, ours being a very old and almost obsolete method.—WEEKLY READER.

MEETINGS FOR THE WEEK.

MONDAY, March 1st.—Society of Arts, 8. (Cantor Lectures). "Industrial Uses of Cellulose," by C. F. Cross, F.C.S.

Society of Chemical Industry, 8. "Relation of Colour to Quality in Malt," by J. W. Lovibond. "Hehner's Bromine Tests for Oils," by J. H. B. Jenkins. "Analysis of Superphosphates," by J. H. Coste.

TUESDAY, 2nd.—Royal Institution, 3. "Animal Electricity," by Prof. A. D. Waller, F.R.S.

Society of Arts, 8. "Gesso," by Matthew Webb.

WEDNESDAY, 3rd.—Society of Arts, 8. "English Orchards," by Geo. Gordon.

Society of Public Analysts, 8. "The Composition of Milk and Milk Products," by H. Droop Richmond. "Estimation of Milk Sugar in Milk" and "Detection of Mixtures of Diluted Condensed or Sterilised Milk with Fresh Milk," by H. Droop Richmond and L. K. Boseley. "Constitution of Milk," by H. Droop Richmond. "Copper in Peas," by R. Bodmer and C. G. Moor, M.A. "Coffee Palace Coffee Infusions," by E. G. Clayton.

THURSDAY, 4th.—Royal Institution, 3. "Greek History and Extant Monuments," by Prof. Percy Gardner, F.S.A.

Society of Arts, 8. "The Mechanical Production of Cold," by Prof. James A. Ewing, M.A., F.R.S.

Chemical, 8. Ballot for Election of Fellows. "Some Hydrocarbons from American Petroleum—1. Normal and Iso-Pentane," by Sydney Young, F.R.S., and G. L. Thomas, B.Sc. "The Vapour Pressures, Specific Volumes, and Critical Constants of Normal Pentane, with a Note on the Critical Point," by Sydney Young, F.R.S. "On the Freezing-point Curves of Alloys containing Zinc," by C. T. Heycock, F.R.S., and F. H. Neville. "The Oxides of Cobalt and the Cobaltites," by A. H. McConnell and E. S. Hanes.

FRIDAY, 5th.—Royal Institution, 9. "Some Curiosities of Vision," by Sheldford Bidwell, F.R.S.

SATURDAY, 6th.—Royal Institution, 3. "Electricity and Electrical Vibrations," by Right Hon. Lord Rayleigh, M.A., F.R.S.

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1945.

THE AGE OF COPPER IN CHALDEA.

By M. BERTHELOT.

THE discoveries made some years ago by M. de Sarzee, at Tello in Chaldea, have brought to our knowledge monuments of a high antiquity, extending back to the origin of civilisation, 5000 or 6000 years ago. They have furnished weapons, ornaments, and tools which throw a new light on the origin of the industry of metals. Such are the objects deposited at the Louvre, which our colleague, M. Heuzey, has kindly referred to my examination. We find here the first and most ancient monuments belonging to the age of copper.

1. I have analysed a colossal lance or blade showing various designs and inscriptions, with the name of a king of Kish which goes back to an epoch anterior to Our-Nina, *i. e.*, about 4000 years before our era. This lance has not been devoted to practical service, but has a hieratic character, having been consecrated to some deity. It is formed of a red metal, being strongly attacked in some parts and changed into a greenish paste.

The filings of the metal consist of copper approximately pure, and I have found in it no tin, zinc, arsenic, or antimony in any appreciable amount.

The oxidised portion consists of hydrated copper oxychloride (atacamite) free from carbonate. There was found in it neither arsenic, antimony, tin, or zinc, but a trace of lead. This substance, after drying in the stove, contained $\text{Cl} = 19.6$.

This oxychloride results from the action of the brackish waters of the soil in the midst of which the blade has been lying for so many centuries. When once the object is brought in contact with the air, the presence of the alkaline chlorides and atacamite threaten a total disaggregation in consequence of its progressive conversion into super-copper oxide. This aggregation results from a certain series of reactions, set up by a small quantity of sodium chloride with the intermediation of atacamite, which I have defined by direct experiment (*Annales de Chimie et Physique*, Series 7, vol. iv., p. 552). Most of the statues of copper found in the same excavations are undergoing this same decomposition at the Museum. They are wrongly labelled "*Objects of Bronze*," as they consist of pure copper.

2. Hatchet with a socket formed of red metal. Broken fragments coated with a greenish patina. A similar instrument is represented in the hands of Chaldean personages on the monuments from the epoch of Our-Nina to that of Goudea, that is, from the year 4000 to the year 3000 before our era.

The fragments which I have analysed consist essentially of metallic copper associated with a little cupric oxide. No tin, lead, zinc, arsenic, or antimony. The hatchet has therefore not been formed of bronze, but of copper sensibly pure.

3. Entire hatchet, red, with a sharp edge, horizontal and socketed. It has been found with its handle below the ancient construction of the king Our-Nina. M. Heuzey regards it as perhaps the most ancient relic met with in these excavations. The metal is hard, of pure copper, free from tin, lead, or zinc, but contains traces of arsenic and phosphorus. It seems to have been hardened by the concurrence of these latter elements. But we do not possess the ores which have served in the manufacture of the Chaldean objects, and we cannot assert, as is the case of the specimens from Sinai, that the presence of arsenic is due to the addition of some substance foreign

to the copper ore properly so called. In any case I must here repeat that we have to do with copper, and not bronze, as the Chaldean tools, &c., contain no tin.

4. Egg-shaped article, of a metallic aspect, weighing 121 grms., found along with the Chaldean remains. The filings consisted of iron partially oxidised, without arsenic, zinc, or alumina.

5. Ingot and filings (ancient) of a white metal, found with the Chaldean remains in an urn of coarse pottery. The filings of the ingot contain:—Silver, 95.1; copper, a small quantity; notable patina; no lead.

6. Leaf of yellow gold, of Chaldean or Assyrian origin. This gold contains neither copper, lead, nor iron in sensible proportion. It contains a considerable quantity of silver, which the minimal weight of the sample at my disposal did not permit me to determine with precision. In this and other cases it is always the antique alloy of gold and silver known under the name of *asem*. The manner of purifying native gold was not well understood in Egypt and Chaldea in those remote ages.

The existence of successive degrees in the use of the purification of the metals, both common and precious, appears from these analyses. In particular, the use of pure copper for arms and tools was common in Chaldea about the year 4000 B.C. It preceded the use of bronze, *i. e.*, copper alloyed with tin, which is found in later articles both in Egypt and Chaldea. We may even add that the form of hatchets with handles, the processes of moulding and manufacture, and even the practical uses to which the tools were destined, have been the same both for the pure hatchets of copper in Chaldea and for the prehistoric hatchets of Europe and Siberia.—*Comptes Rendus*, cxxiv., p. 328.

CARBIDE OF CALCIUM.

In consequence of the growing importance of carbide of calcium, and the fact that the mere contact of moisture with this material causes a dangerous evolution of the highly inflammable gas known as Acetylene, the Home Secretary has caused inquiries to be made into the subject, with the result that an Order in Council has to-day been made under the 14th Section of the "*Petroleum Act, 1871*," bringing carbide of calcium within the operation of that Act.

Accordingly, from the date on which such Order comes into force, *viz.*, 1st April, 1897, it will be unlawful to keep carbide of calcium except in virtue of a license to be obtained from the Local Authority under the "*Petroleum Act*."

Any Local Authority to whom application may be made for a license to keep carbide of calcium can, if it so desires, obtain, on application to the Home Office, a Memorandum showing the character of the risks to be guarded against, and containing suggestions as to the nature of the precautions likely to be most effectual for securing safety.

Whitehall, 26th February, 1897.

DETERMINATION OF SULPHUR IN IRONS.

By OTTO HERTING.

In the laboratories of many iron works it is customary to be content with Wiborgh's method (colorimetric), as the determination by the bromine method is tedious, and very unpleasant where the ventilation arrangements are imperfect. I will by no means deny the value of the colorimetric method, but wish to point out that the results are frequently very inaccurate, owing to imperfectly constructed apparatus or to escapes at the joints. The latter

we may best detect by means of nitro-prusside papers. A disadvantage in Wiborgh's method which cannot be overlooked is, that we are compelled to use a very small quantity of material (0.2 to at most 0.8 grm.) of borings, which is far too small a quantity. The most expeditious quantitative method which yields perfectly satisfactory results, is, in my opinion, the following, which E. F. Wood (of the Homestead Steel Works) publishes briefly in Blair's "Analysis of Iron" p. 71:—5 or 10 grms. of borings are treated with hydrochloric acid; the hydrogen sulphide evolved is conducted into an ammoniacal cadmium salt (acetate or chloride); the cadmium sulphide is collected on a small filter, shaken out with cold water in an Erlenmeyer flask, mixed with 1/20 solution of iodine in excess, which is then titrated back with a corresponding solution of thiosulphate. The entire operation can be completed in one hour.

About two years ago Prof. de Koninck proposed to determine sulphur in irons, adding a small quantity of stannous chloride to the hydrochloric solution to prevent the formation of ferric chloride. I am now of De Koninck's opinion, that the oxygen of the air in 1 litre does not react so quickly upon hydrogen sulphide as to occasion a separation of sulphur which might thus escape determination. Hence it is not necessary to expel the air of the flask by the introduction of a current of hydrogen or carbon dioxide.—*Chemiker Zeitung*, Feb. 6.

THIRD ANNUAL REPORT OF COMMITTEE ON ATOMIC WEIGHTS.

RESULTS PUBLISHED DURING 1895.*

By F. W. CLARKE.

(Concluded from p. 101.)

Helium and Argon.—The true atomic weights of these remarkable gases are still in doubt, and so far can only be inferred from their specific gravities. For argon, the discoverers, Rayleigh and Ramsay (*Phil. Trans.*, clxxvi., 220—223) give various determinations of density, ranging $H=1$) from 19.48 to 20.6. The value 19.9 they regard as approximately correct.

For helium, Ramsay (*Journ. Chem. Soc.*, iii., 684) gives the density 2.18, while Langlet (*Ztschr. Anorg. Chem.*, x., 289) finds the somewhat lower value 2.00.

From one set of physical data both gases appear to be monatomic, but from other considerations they are supposable diatomic. Upon this question, controversy has been most active, and no final settlement has yet been reached. If diatomic, argon and helium have approximately the atomic weights 2 and 20 respectively; if monatomic, these values must be doubled. In either case helium is an element lying between hydrogen and lithium; but argon is most difficult to classify. With the atomic weight 20, argon fills in the eighth column of the periodic system, between fluorine and sodium; but if it is 40, the position of the gas is anomalous. A slightly lower value would place it between chlorine and potassium, and again in the eighth column of Mendeleeff's table, but for the number 40 no opening can be found.

It must be noted that neither gas, so far, has been proved to be absolutely homogeneous; and it is quite possible that both may contain admixtures of other things. This consideration has been repeatedly urged by various writers. If argon is monatomic, a small impurity of greater density, say of a unknown element falling between bromine and rubidium, would account for the abnormality of its atomic weight, and tend towards the reduction of the latter. If the element is diatomic, its classification is easy enough on the basis of existing data. Its resemblance

	H = 1.	O = 16.
Aluminum	26.91	27.11
Antimony	119.52	120.43
Argon	?	?
Arsenic	74.52	75.09
Barium	136.40	137.43
Bismuth	206.54	208.11
Boron	10.86	10.95
Bromine	79.34	79.95
Cadmium	111.08	111.93
Cæsium	131.89	132.89
Calcium	39.78	40.08
Carbon	11.92	12.01
Cerium	139.1	140.2
Chlorine	35.18	35.45
Chromium	51.74	52.14
Cobalt	58.49	58.93
Columbium	93.3	94.0
Copper	63.12	63.60
Erbium	165.0	166.3
Fluorine	18.89	19.03
Gadolinium	154.9	156.1
Gallium	68.5	69.0
Germanium	71.75	72.3
Glucinium	9.01	9.08
Gold	195.74	197.24
Helium	?	?
Hydrogen	1.00	1.008
Indium	112.8	113.7
Iodine	125.89	126.85
Iridium	191.66	193.12
Iron	55.60	56.02
Lanthanum	137.6	138.6
Lead	205.36	206.92
Lithium	6.97	7.03
Magnesium	24.11	24.29
Manganese	54.57	54.99
Mercury	198.5	200.0
Molybdenum	95.26	95.98
Neodymium	139.4	140.5
Nickel	58.24	58.69
Nitrogen	13.94	14.04
Osmium	189.55	190.99
Oxygen	15.879	16.00
Palladium	105.56	106.36
Phosphorus	30.79	31.02
Platinum	193.41	194.89
Potassium	38.82	39.11
Praseodymium	142.4	143.5
Rhodium	102.23	103.01
Rubidium	84.78	85.43
Ruthenium	100.91	101.68
Samarium	148.9	150.0
Scandium	43.7	44.0
Selenium	78.4	79.0
Silicon	28.18	28.40
Silver	107.11	107.92
Sodium	22.88	23.05
Strontium	86.95	87.61
Sulphur	31.83	32.07
Tantalum	181.2	182.6
Tellurium	126.1?	127.0?
Terbium	158.8	160.0
Thallium	202.60	204.15
Thorium	230.87	232.63
Thulium	169.4	170.7
Tin	118.15	119.05
Titanium	47.79	48.15
Tungsten	183.44	184.84
Uranium	237.77	239.59
Vanadium	50.99	51.38
Ytterbium	171.7	173.0
Yttrium	88.28	88.95
Zinc	64.91	65.41
Zirconium	89.9	90.6

* Read at the Cleveland Meeting, December 31, 1895. From the *Journal of the American Chemical Society*, xviii., No. 3.

to nitrogen, as regards density, boiling-point, difficulty of liquefaction, &c., lead me personally to favour the lower figure for its atomic weight, and the same considerations may apply to helium also. Until further evidence is furnished, therefore, I shall assume the values 2 and 20 as approximately true for the atomic weight of helium and argon.

Carbon.—Wanklyn (CHEM. NEWS, lxxii., 164; see also *Phil. Mag.*, August, 1895; also the reports of this committee for 1893 and 1894), on the basis of his investigations into the composition of hydrocarbons, reiterates his belief that the atomic weight of carbon is not 12 but 6. This question is one which falls rather outside the scope of this report and needs no further discussion here. If Wanklyn's contention is sustained, the value assigned to carbon in the table accompanying this paper should be divided by two.

In the accompanying table of atomic weights, the values are given according to both standards, $H=1$ and $O=16$. Many of the figures are the results of new and complete re-calculation from all available data, made in the preparation of a new edition of my "Re-calculation of the Atomic Weights."

ELECTRIC SHADOWS AND LUMINESCENCE.*

By Prof. SILVANUS P. THOMPSON, D.Sc., F.R.S., M.R.I.

(Continued from p. 106).

AND now let me remark that not one of all the tubes shown since the first one is capable of showing a shadow upon the fluorescent screen outside, or of taking a photograph through a sheet of aluminium. Even the brilliant tube which showed so excellently the shadow of the cross, fails to show any result after hours of vain waiting. It yields no rays that will penetrate aluminium. For experiments with Röntgen rays it is absolutely necessary that the process of exhaustion should be carried beyond the stage that suffices for the production of cathode shadows; it must be pushed to about that limit which Crookes himself described as his unit for the degree of vacuum, namely, one-millionth of an atmosphere. I do not say that with long exposures photographs cannot be taken when the degree of exhaustion is lower. Something depends, too, upon the degree to which the electric discharge is stimulated, and something also depends upon the shape and structure of the tube and upon the size and shape of the cathode. But on none of these things does the emission of X-rays depend so much as upon the degree of vacuum. The highly exhausted vacuum is the one real essential.

So soon as Crookes's researches upon electric shadows had become known, electricians set to work to try to produce electric shadows in ordinary air without any vacuum. One of the ablest of experimenters, Prof. W. Holtz, was successful, using as a source of electric discharge the electrified wind which is given off by a metal point attached to the pole of an influence machine. If in a perfectly dark room such a point is placed opposite and at a few inches from a wooden disc covered with white silk and connected at its back or edges to the other pole of the machine, it will be observed to show a pale luminosity over a circular patch where it is struck by the electric wind. If then the object is brought between the disc and the point a shadow will be observed to be cast upon the white surface. Non-conductors do not cast shadows as well as conductors do. A piece of thin mica scarcely casts a shadow at all until it is moistened. Double shadows can be got by using two disks covered with silk facing one another; any conducting object introduced between them casts a shadow on both. If such a shadow

from an electrified point is cast downward upon a sheet of ebonite or pitch, the parts not shaded are found afterwards to remain electrified, and can be discovered by scattering over them Lichtenberg's mixed powders of red-lead and lycopodium, thus perpetuating the shadow.

But now it is possible to produce electric shadows in another way, photographically, as has been known for some years (*Proc. Phys. Soc. Lond.*, xi., 353, 1892), from metal objects such as coins, by simply laying them down upon a photographic dry-plate (a gelatino-bromide plate) and sending an electric spark (from an induction-coil) into them.

Fig. 3 shows the arrangement adopted by the Rev. F. J. Smith, who is kind enough to exhibit in the library to-night some scores of his beautiful "inductoscript" photographs. Upon the screen I throw a few samples, including a print of one of the Jubilee coins (Fig. 4). These curious photographs are produced simply by the chemical action of the electric discharges which stream off from all the projecting portions, and so roughly reproduce an image of the coin. Since Röntgen's discovery many persons have announced their supposed discovery of the production of electric shadow-pictures without the aid of a Crookes tube. What they have really observed is, however, totally different. They have not been producing X-rays at all, but have merely re-discovered these inductoscript shadows.

Between the researches of Crookes, however, and those of Röntgen, there came in a very remarkable body of researches in Germany. I have but to name Goldstein, Puluj, Hertz, Wiedemann, and Lenard (See NOTE), amongst the workers, to show what interest has been concentrated on the subject. Hertz, whose loss Science has not ceased to lament, observed that a part at least of the cathode rays were capable of passing through thin aluminium sheet, a property which confirmed him in his previous doubt as to the material nature of the cathodic discharge. His pupil, Philipp Lenard, now Prof. Lenard, of Aachen, took up the point. He fitted up a tube with a small window of aluminium foil opposite the cathode, its form being that shown in Fig. 5. The cathode was a flat disk on the end of a glass-covered wire stem. The anode was a cylindrical tube of brass surrounding the cathode. Upon the further end of the tube a brass cap was fixed by means of vacuum-tight cement. Over a small orifice in this brass cap was set the aluminium window of foil only 1-400th m.m. thick. By this means he was able to do what had previously been supposed impossible—bring the cathode rays out into the open air. Or, at least, that is what he appears to have considered that he was doing. Certainly he succeeded in bringing out from the vacuum tube rays that, if not actual prolongations of the cathode rays, were closely identified with them. He examined their properties both in the open air and in gases contained in a second chamber beyond the window, and found them to be capable of producing photographic impressions on sensitive plates. He further examined the question whether they can be deflected by a magnet. Fig. 6, which is copied from Lenard's paper, shows the results. The row of spots on the left side shows the photographic effect under various different conditions of experiment when there was no magnet present. The spots in the right-hand row show the effects obtained when a magnet was present. For example, in the third row from the top it is seen that the bundle of rays when subjected to the influence of the magnet is partially dispersed, the spot being enlarged sideways and having a kind of nebulous tail. This proves that through the aluminium window there came some rays which were deflected by a magnet, and some rays also which were not deflected by a magnet. The question naturally arises whether the rays which Lenard had thus succeeded in bringing out into the open air are the same thing as the rays with which Crookes had been working with inside the vacuum. To that question the final answer cannot yet be given. Certainly some of the Lenard rays resemble the interior cathode rays; but some

* A Lecture delivered at the Royal Institution of Great Britain, Friday, May 8, 1896.

differ in the crucial respect of deflectability by the magnet. The higher the degree of vacuum, the less are the rays deflected.

NOTE.

Goldstein, in his "Researches on the Reflection of Electric (*i. e.*, Kathode) Rays," in *Wiedemann's Annalen* (xv., 246, 1882), came very near to the discovery of the Röntgen rays. After pointing out that Hittorf had held the opinion that the kathode rays end at the place where

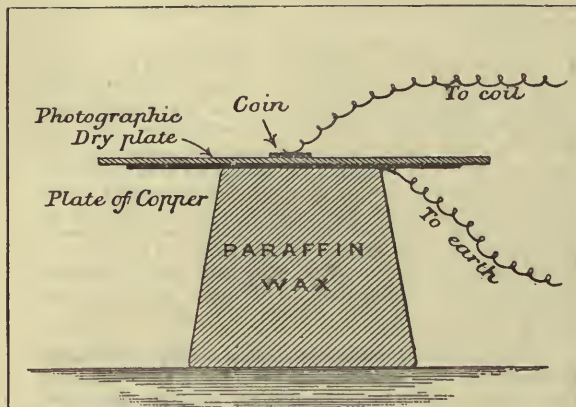


FIG. 3.

they strike upon a solid wall, and that they are unable to proceed in any direction at all from thence, Goldstein directs attention to the circumstance that fluorescent patches are sometimes seen at the end of crooked tubes, where they could not have been caused by the direct impact of kathode discharges. He discusses the question whether this is due to reflection or to a deflection caused by the spot where impact first took place having become electrified negatively, and therefore acting as a secondary kathode. The latter hypothesis is rendered untenable by his observation that if the spot of first impact is made an anode the effect still occurs. He then shows that the phenomena are inconsistent with a specular reflection, but



FIG. 4.

are explained by supposing that there is a diffuse reflection. He then sums up as follows:—"A bundle of kathode rays does not end, at least under those circumstances under which it excites phosphorescence, at the place where it strikes upon a solid wall, but from the place of impact on the wall there proceed electric rays in every direction in the gaseous space. These rays may be considered as reflected. Any solid wall of any property whatever may serve as a reflecting surface. It is immaterial whether or not it is capable of phosphorescence, or whether it consists of an insulator or of a conductor.

The reflection is diffuse, no matter whether the surface is dull or most highly polished. An anode reflects the kathode rays sensibly as well as a neutral conductor or an insulator. The reflected rays have, like the direct kathode rays, the property to excite phosphorescence at their ends. They are subject to deflection, and their ends are deviated in the same sense as the ends of kathode rays, which would extend from the reflecting surface toward the place hit by the reflected rays."

Puluj, "Radiant Electrode Matter, and the so-called Fourth State." Published in vol. i. of "Physical Memoirs," by the Physical Society of London, 1889. These are translated from papers published in 1883 in the *Memoirs of the Imperial Academy of Sciences at Vienna*.

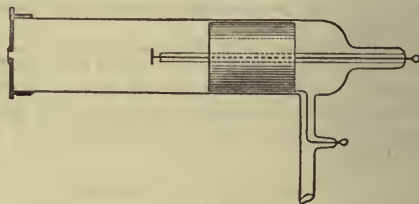


FIG. 5.

H. Hertz. "Researches on the Glow-Discharge," *Wied. Ann.*, xix., 782, 1883. Hertz regards the kathode rays as a property of the ether, not as consisting of moving particles. He finds the kathode rays to consist of a heterogeneous variety of kinds which differ from one another in their properties of causing phosphorescence, of being absorbed, and of being deflected by the magnet.

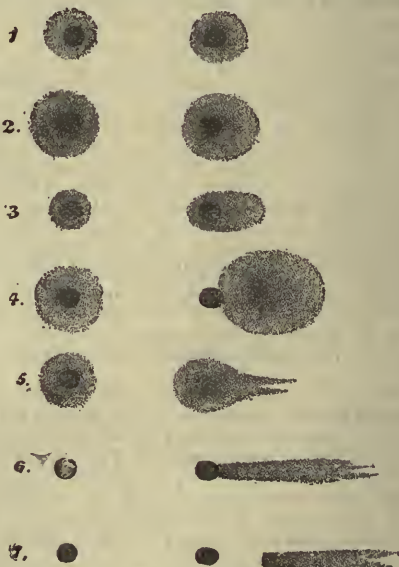


FIG. 6.

"On the Transmission of the Kathode Rays through Thin Layers of Metal" (xiv., 28, 1892), Hertz finds that glass fluoresces in kathode rays, even if covered with gold-leaf or thin films of various metals, though not if covered with thin mica. Aluminium was found best, and allowed fluorescence to occur even when a sheet of aluminium-leaf was used so thick as to be opaque to light. A diaphragm of thin aluminium-leaf on a metal frame placed inside a Crookes tube at 20 c.m. from the kathode, permitted enough rays to pass to give a tolerably bright and even fluorescence over the whole of the further end of the tube. These rays, after passing through the leaf

of metal, still showed rectilinear propagation (with some diffusion), and had not lost the property of being deflected by the magnet.

E. Wiedemann's papers, which are of special importance, have mostly appeared in *Wiedemann's Annalen*. The following are the chief of them. Some of the later have been written in collaboration with Prof. H. Ebert.

"On the Phosphorescent Light excited by Electric Discharges" (*Wied. Ann.*, ix., 157, 1880).

"On Electric Discharges in Gases" (xx., 756, 1881).

"On Fluorescence and Phosphorescence," Pt. I (xxxiv., 446, 1888).

"On the Mechanism of Luminosity" (xxxvii., 177, 1889).

"On Kathodo- and Photo-Luminescence of Glasses" (xxxviii., 488, 1889).

"On Electric Discharges in Gases and Flames" (xxxv., 209, 220, 234, 237, 255, 1888).

"On Electric Discharges" (xxxvi., 643, 1889).

"On the Apparent Repulsion of Parallel Kathode Rays" (xli., 158, 1892).

"On Electric Discharges; Excitation of Electric Oscillations and the Relation of Discharge-tubes to the same" (xlviii., 549, and xlix., i, 1893).

"Researches on Electrodynamical Screening-action and Electric Shadows" (xlix., 32, 1893).

"Luminous Phenomena in Electrode-less Rarefied Spaces under the Influence of Rapidly-alternating Electric Fields" (l., i, 221, 1893).

With J. B. Messerschmitt, "On Fluorescence and Phosphorescence, Pt. II., Validity of Talbot's Law" (xxxiv., 463, 1888).

With H. Ebert, "On the Transparency of Kathode Deposits," *Sitzber. d. Phys.-Med. Soc. zu Erlangen*, Dec. 14, 1891.

Lenard's Papers are:—

"Note on a Phosphoroscope, with Spark Illumination" (*Wied. Ann.*, xxxiv., 918, 1888).

With M. Wolf, "Luminescence of Pyrogallol Acid" (xxxiv., 918, 1888).

With V. Klatt, "On the Phosphorescence of Copper, Bismuth, and Manganese in the Sulphides of Alkaline Earths" (xxxviii., 90, 1889).

"On Kathode Rays in Gases at Atmospheric Pressure, and in the most Extreme Vacuum" (li., 225, 1894).

"On the Magnetic Deflexion of the Kathode Rays" (lii., 22, 1894).

"On the Absorption of the Kathode Rays" (lvi., 255, 1895).

(To be continued).

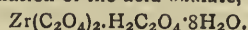
THE OXALATES OF ZIRCONIUM.

By F. P. VENABLE and CHARLES BASKERVILLE.

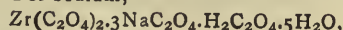
THE text-books of chemistry make either very little or no reference to the oxalates of zirconium. Beyond an occasional reference to the oxalate or basic oxalate gotten by precipitating with oxalic acid or an oxalate, we can find little mention of these compounds. Behrens, in his microchemical work, speaks of an oxalate prepared as colourless pyramids by precipitating a solution of zirconium sulphate with potassium binoxalate, but no analyses are given, and the crystals could scarcely have been the pure oxalate. Paykull (*Ofv. af. Vet. Ak. Förhandl.*, ref. in *Ber. d. Chem. Ges.*, xii., 1719) speaks of double oxalates being prepared with the alkaline oxalates (1 : 2) and his failure to prepare the neutral oxalate. His methods, and indeed full results, are unknown to us, as we did not have access to the original paper.

We may summarise the work which follows in the succeeding pages by saying that we found it possible to prepare the basic oxalates by precipitation. This was

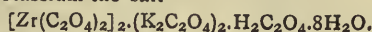
usually in the form of $\text{Zr}(\text{C}_2\text{O}_4)_2 \cdot \text{Zr}(\text{OH})_4$, though other ratios were gotten. The neutral oxalate we did not succeed in preparing, but instead the tendency seems to be toward the formation of the acid oxalate,—



This tendency toward the formation of acid salts was shown also in the double oxalates. Two of these were prepared. For sodium,—



and for potassium the salt—



The oxalate with ammonium as a constituent was not so easy of preparation in a pure state. The compound secured was $\text{Zr}(\text{C}_2\text{O}_4)_2 \cdot 2(\text{NH}_4)_2\text{C}_2\text{O}_4$. The experiments and analyses are given in detail.

Zirconium Oxalates.

The Oxalate Gotten by Precipitation.—On the addition of a saturated solution of oxalic acid to a slightly acid solution of zirconium chloride until no further precipitation occurred, a gelatinous precipitate formed which had very nearly the composition $\text{Zr}(\text{C}_2\text{O}_4)_2 \cdot 2\text{Zr}(\text{OH})_4$. Analysis I. gave Zr 46.39, and C_2O_4 30.89, instead of the theoretical 46.40 and 30.93 respectively. The filtrate from this was turbid, and on standing yielded another precipitate which had nearly the composition $2\text{Zr}(\text{C}_2\text{O}_4)_2 \cdot 3\text{Zr}(\text{OH})_4$.

These basic oxalates are very difficultly soluble in acids, and of extremely fine subdivision, settling slowly and passing through even the best filters. It does not seem probable that they could be secured of very constant composition. Probably basic oxalates with many different ratios between the oxalate and the hydroxide might be secured. On drying at 100°, or even a little lower, the oxalic acid is gradually volatilised and lost. This is true of all the oxalates and double oxalates prepared, so that the only mode of drying these preparations was between filter-paper.

The Acid Oxalate prepared by Crystallisation.—In preparing this oxalate, zirconium hydroxide was dissolved in oxalic acid. The hydroxide is quite soluble in oxalic acid, and a concentrated solution is readily obtained. A considerable excess of the acid is required to hold the oxalate thus formed in solution. If this solution be acidified by means of hydrochloric acid a very fine precipitate is obtained, settling very slowly, easily passing through the best filter-papers, and insoluble even in a considerable excess of the acid, but soluble in concentrated sulphuric acid. This precipitate was not analysed, nor were the exact conditions of its formation determined, as its examination did not promise results of sufficient importance to justify overcoming the difficulties in the way.

On evaporating the acid solution of the oxalate the excess of oxalic acid first crystallises out. In the various preparations made, the first one or two crops of long crystals were found to be nearly pure oxalic acid, and were rejected. Then the form of the crystals changed to small granular or prismatic masses, and with each succeeding crop of crystals the percentage of zirconium increased, reaching speedily an approximately constant ratio. No difference in the form of the crystals in these different crops could be detected on superficial examination, and hence it was impossible to distinguish between the zirconium oxalate and the oxalic acid almost free of zirconium, except by analysis. In no case was the normal oxalate secured. The analyses showed a tendency toward the formation of an acid oxalate and to mixtures of this with the normal oxalate. These mixtures were gotten in the later crystallisations, but the last crystallisation, when nearly the whole would solidify into a crystalline mass, showed decreased percentages of zirconium. It is possible that larger amounts than we had at our disposal would enable one so to fraction the crystallisation as to secure a pure oxalate. It is, however, questionable

whether the normal oxalate can exist in solution without admixture with some oxalic acid.

Four series of crystallisations were made, and in two cases fairly abundant crops of crystals corresponding to the acid oxalate were obtained. In each series enough of the zirconium hydroxide was taken to form about 20 grms. of the oxalate.

	First series. Sixth fraction. II.	Second series. Fifth fraction. III.	$Zr(C_2O_4)_2 \cdot H_2C_2O_4$.
Zr	25'44	25'28	25'53
C_2O_4	74'55	74'72	74'47

These are calculated upon the water-free basis. The crystals contained 29'34 and 29'27 per cent of water respectively, where the salt $Zr(C_2O_4)_2 \cdot H_2C_2O_4 \cdot 8H_2O$ contains 28'90 per cent. Other crops of crystals contained percentages of zirconium not varying greatly from these given above as 28'14, 27'62, 24'9, 23'83. The percentage of zirconium in the normal oxalate is 33'96.

Zirconium Sodium Oxalate.

The addition of sodium oxalate to a slightly acid solution of zirconium chloride gives a gelatinous white precipitate. Most of this dissolves in an excess of the oxalate. The undissolved portion settles to the bottom, and after prolonged standing a second layer of a more powdery appearance forms. This can also be gotten by concentration of the filtrate from the first precipitate. Analysis showed that the first gelatinous precipitate was chiefly $Zr(OH)_4$. The second precipitate was a double oxalate of zirconium and sodium, but was either of inconstant composition (varying ratios of sodium to the zirconium), or was decomposed by the washing.

The analyses, calculated on the dry basis, gave:—

	IV.	V.	VI.
Zr	53'12	46'86	41'98
Na	9'16	4'10	1'07
C_2O_4	38'06	39'64	42'95

If the solution made with the excess of sodium oxalate was diluted considerably with water, a gelatinous precipitate was formed, very fine and insoluble. Precipitates were also formed by the addition of hydrochloric acid. This mode of forming the double oxalate was abandoned, and the following method was adopted with greater success. Zirconium hydroxide was dissolved in an excess of oxalic acid, and to this a concentrated solution of sodium hydroxide was added, bringing it nearly to neutralisation. When the solution was concentrated, an abundant crop of crystals was obtained on cooling, a good deal of heat being evolved in the mixing. Further evaporation yielded other crops of crystals. These were washed, dried between filter-paper, and analysed. The results are given in the following table:—

	VII.	VIII.	IX.	Calculated.
Na	18'14	17'46	17'75	18'19
Zr	12'59	12'66	12'78	11'93
C_2O_4	69'27	66'89	69'47	69'88

These results show a somewhat wide variation from those calculated. This probably arises from the fact that the fractions were not composed of the crystals of a single kind of oxalate, but had other oxalates mixed with them in small amounts. Examined under a magnifying glass they seemed to be homogeneous, but the different crops could not be distinguished from one another. They were all small, hard prismatic crystals, somewhat difficultly soluble in water. One set of crystals, the analysis of which is reported under VII. in the above table, was re-dissolved in water and re-crystallised. On analysis it yielded the following results:—

	VII.	XI.
Na	18'14	18'19
Zr	12'59	12'71
C_2O_4	69'27	69'10

These were calculated upon a water-free basis. The crystals from the various crops mentioned above did not contain a very constant amount of water, but ranged from 9'13 to 11'06. The calculated amount of water in $Zr(C_2O_4)_2 \cdot 2Na_2C_2O_4 \cdot H_2C_2O_4 \cdot 5H_2O$ is 10'62. It would seem, therefore, that the tendency, when this method of formation is adopted, is toward the formation of crystals containing free oxalic acid and with the sodium and zirconium oxalates bearing a ratio of three to one.

Zirconium Potassium Oxalate.

The curdy precipitate gotten by precipitating zirconium chloride with normal potassium oxalate is insoluble in an excess of either of the substances. The precipitate first obtained is an impure zirconium hydroxide, containing only small amounts of oxalic acid. The supernatant liquid, on concentration, yields needle-like crystals of potassium oxalate, carrying only traces of zirconium. After the separation of a good deal of this potassium oxalate, further concentration yielded a gelatinous substance having the composition (XII.): Zr, 39'34; K, 5'06; C_2O_4 , 43'05; which seems to be a basic zirconium oxalate, mixed or united with a small proportion of potassium oxalate. If the potassium be calculated as potassium oxalate and subtracted, the composition of the remainder would be approximately $Zr(OH)_4 \cdot Zr(C_2O_4)_2$.

On adding potassium binoxalate to a solution of zirconium chloride a white curdy precipitate was obtained which was not completely soluble in excess of the binoxalate. The somewhat turbid solution was filtered and evaporated. Large crystals resembling those of oxalic acid formed. These were separated, and on analysis proved to be oxalic acid. At the same time a number of small crystals were formed, which were mechanically separated, washed, and dried. These were analysed, and are reported under XIII. A further crop was gotten from the mother-liquor, and the analysis is given under XIV.

	XIII.	XIV.
Zr	19'59	17'99
K	16'18	13'91
C_2O_4	64'23	68'09

The curdy precipitate which first formed was also examined, and found to have the composition $Zr(C_2O_4)_2 \cdot 2Zr(OH)_4$.

The addition of a solution of potassium tetroxalate to zirconium chloride gave a gelatinous precipitate of zirconium oxalate (basic), carrying a little potassium oxalate. Subtracting the potassium oxalate, the percentages (XVI.) Zr 39'09, and C_2O_4 38'63, are left, which are not very different from the figures gotten for the precipitate from potassium oxalate (neutral).

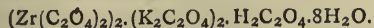
This curdy gelatinous precipitate was dissolved in excess of tetroxalate, and the solution placed over sulphuric acid to crystallise, and yielded crystals having the composition (XVII.): Zr 20'85, K 16'72, and C_2O_4 62'31. As will be seen, these are not far from the 1 : 2 zirconium potassium oxalate, with excess of oxalic acid.

When potassium hydroxide was added to a solution of zirconium oxalate in oxalic acid until nearly neutral, and then set aside for crystallisation, various crops of crystals were gotten, as in the case of the double sodium oxalates. These crops of crystals were similar in appearance to the sodium crystals. They were analysed and showed fairly constant composition.

	XVIII.	XIX.	XX.	XXI.	$(Zr(C_2O_4)_2)_2 \cdot (K_2C_2O_4)_2 \cdot H_2C_2O_4$
Zr..	18'08	19'25	19'83	18'47	18'95
K ..	16'41	16'35	14'84	14'46	16'34
C_2O_4	66'51	64'40	65'33	67'07	64'71

The three previous analyses may also be referred to here as having approximately the same composition. (See Analyses XIII., XIV., XVII.). These are calculated as water-free. In the Analyses XVIII. and XIX. the per-

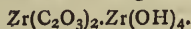
centages of water were 12.99 and 12.38. These would correspond to the formula—



In this case, as in the zirconium oxalates and the sodium oxalates, the crystals seem to form only along with free oxalic acid, giving acid salts.

Zirconium Ammonium Oxalates.

The addition of a solution of ammonium oxalate to the slightly acid solution of zirconium chloride gave a heavy gelatinous precipitate which was soluble in excess of ammonium oxalate, and proved to be zirconium hydroxide with more or less zirconium oxalate and small amounts of ammonia. The filtrate from this precipitate was evaporated slowly and a fine crystalline powder obtained. This contained (XXII.) Zr 42.17 per cent, and C_2O_4 39.86 per cent. This is in fair agreement with—



When ammonium oxalate is added until the first gelatinous precipitate is re-dissolved, and then evaporated to crystallisation, different crops of crystals can be gotten containing various amounts of ammonia. These did not seem to have any regular composition in our experiments, and were looked upon as basic zirconium oxalates with varying amounts of ammonium oxalate present. Thus for one of these the figures (XVIII.) Zr 31.48, NH_3 7.14, and C_2O_4 61.38 were gotten.

Abandoning this method, and using the one adopted in the cases of the sodium and potassium double oxalates, a more favourable result was obtained. Zirconium hydroxide was dissolved in excess of oxalic acid, and then this was nearly neutralised by means of ammonium hydroxide. Analyses of these crops of crystals follow:—

	XXIV.	XXV.	$\frac{\text{Zr}(\text{C}_2\text{O}_4)_2}{2(\text{NH}_4)_2\text{C}_2\text{O}_4}$
Zr	16.55	16.66	17.58
NH_3 ..	14.46	13.35	13.28
C_2O_4 ..	69.99	69.99	68.94

While these do not show that the crystals had been thoroughly purified, the results indicate that the composition is one zirconium oxalate to two ammonium oxalate. On re-crystallising one of these crops of crystals, zirconium hydroxide was observed to separate when the solution was heated (to evaporate to crystallisation), and the crystals which were obtained consisted of ammonium oxalate alone.

In general it may be stated that the zirconium oxalate fails to show any decided tendency to enter into clearly-defined combinations with the alkaline oxalates, exhibiting rather a power of crystallising along with them in mixtures of any proportions. It can only be said at best that under the conditions of our experiments certain ratios seem to be preferred, and appeared more persistently. In all cases the crystals formed from oxalic acid solutions, and this free oxalic acid crystallised with them, giving acid oxalates.—*Journ. Amer. Chem. Soc.*, xix., p. 12.

Royal Institution. — A General Monthly Meeting of the Members of the Royal Institution was held on March 1st, Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, presiding. The following were elected Members:—F. J. Beaumont, Major C. T. Blewitt, R.A., J. F. L. Brunner, James Cadett, J. C. Carter, John Cohen, Mrs. Thomas Collier, I. G. Craggs, T. Donaldson, Henry Edmunds, Mrs. Henry Edmunds, G. S. Elliot, W. A. Frost, F.R.C.S., W. T. Garnett, J.P., H. A. Harben, Dr. F. Hewitt, F. W. Hildyard, Mrs. George King, H. Leitner, the Rev. J. D. Parker, E. M. Preston, J. M. Richards, Colonel G. Sartorius, F. H. Schwann, Dr. W. R. Smith, H. A. Stern, C. J. Stewart, G. L. Stewart, Mrs. A. D. Waller, and Mrs. J. Lawson Walton.

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, February 26th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

Mr. J. H. VINCENT read a paper on the "Photography of Ripples."

If mercury is used as the medium, all waves less than 1.3 c.m. long come under Lord Kelvin's definition of a ripple; that is to say, they are waves whose lengths are less than such as are propagated with minimum velocity. Vibrations in mercury of about 200 per second and upwards generate waves whose propagation is controlled almost entirely by surface tension, and these waves are therefore classed as "capillary ripples." Their speed of propagation is of the order of about one foot per second. They are invisible, owing to their high frequency, not in consequence of the velocity of their propagation. It is usual to examine them by some stereoscopic method.

Mr. Vincent obtains photographs of the disturbed mercury surface by the sudden illumination of an electric spark. The spark is about half a centimetre in length, and it lasts about one two-hundred-thousandth part of a second. Its brightness is increased by an auxiliary spark-gap. The optical arrangement consists of two lenses, one in the path of the incident light, and another to converge the reflected light from the mercury surface into a photographic camera. Ripples are set up in the mercury by a stylus attached to a tuning-fork. For this purpose it is generally sufficient to give a slight blow to the prongs; but when continuous vibration is required the tuning-fork can be connected by a thread to an electrically driven fork, as suggested by Mr. Watson.

The first photograph shows a series of circular waves, set up by a single stylus attached to a fork vibrating 180 times a second. Fixed points at known distance, just above the mercury surface, enable the wave-lengths to be deduced from the photographs; and, as the frequency is known, the surface tension may be easily calculated.

In a second photograph, two styluses are attached to the same prong. Dark lines are seen to radiate from the region between the centres of oscillation; these are the lines of minimum disturbance—hyperbolae, of which the centres of disturbance are the foci. This photograph illustrates "interference" similarly to the optical method of Young and Fresnel.

A third photograph shows the formation of elliptical curves of disturbance, being the loci of the intersection of two series of circles, corresponding one to each of the two centres of vibration. Unlike the system of hyperbolae, these ellipses are not at rest, but travel outward from the sources. In order to render these ellipses stationary, it would be necessary to change one of the sources into a sink, towards which the circular waves might converge; the photograph would then correspond to the optical device of M. Meslin, who obtains interference fringes by means of a screen placed between two point centres, one a source, and the other a sink.

The phenomena of interference and diffraction are well shown in a photograph of a point source and a reflecting line. The reflector here is one side of a triangular piece of microscope cover-glass. The interference lines are due to the mutual action of incident and reflected rays; they are analogous to Lloyd's single-mirror fringes. Other photographs exhibit analogues of "spherical aberration" and "forced vibration."

Mr. Vincent acknowledged his indebtedness to Mr. Boys for the recommendation of attempting the photography of capillary ripples.

Mr. Boys congratulated the author upon the way in which the experimental difficulties had been overcome. The results would bear a good deal of close examination, and they would be found to present analogues of the

greatest service in demonstrating the phenomena of acoustics and optics. Such photographs were far better than geometrical pictures drawn by instruments. For example, in the photograph illustrating the regions of minimum disturbance by lines radiating from a two-point source, it was easy to make out the positions where the two series of waves were half a period behind one another. The crests and troughs appeared as a set of dark and light concentric alternating circles, broken up into short arcs by radiating lines—the loci of minimum disturbance; all the crests on one side of any particular radiating line were seen to correspond to troughs on the other side, so that the field of disturbance was mapped out as in acoustics. One set of phenomena yet awaited illustration by this photographic method, and that was "diffraction" from a grating. It might be possible to use as an exciter a comb with chisel-shaped points. He did not think it would be possible to go quite so far as to reproduce analogues of spectral analysis. Since wavelength varies with surface tension, it was possible to vary the wave-length by dropping a little ox-gall or soap solution upon the mercury surface.

Mr. BLAKESLEY asked why no reflections occurred from the sides of the mercury retainer.

Mr. BOYS said the waves were lost at the edges of the meniscus. The mercury was kept in position by an angular ring of thin glass.

Mr. APLEYARD suggested that the analogue of refraction might be obtained by an alteration of the surface-tension over a small area by amalgamation or other means.

Mr. VINCENT thought this could be done, but that it would be very difficult.

The PRESIDENT proposed a vote of thanks to the author.

Mr. ELDER then read a paper by Mr. BECKIT BURNIE on "*The Thermo-electric Properties of some Liquid Metals.*"

The investigation was made with a view to determining the effect of melting upon the thermo-electric properties of certain metals. The metal to be tested is contained in a W-shaped glass tube, of which one limb can be cooled and the other heated. Thus one limb can contain molten, and the other solid, metal. Copper wires are plunged one into each limb, and through these connection is made with a galvanometer. The thermal junctions, therefore, are copper-hot metal and copper-cold metal. The temperature is deduced from a separate thermal couple, calibrated by a mercurial thermometer. Curves are drawn co-ordinating temperature and electromotive force. It is found that their slope depends upon the rate of cooling or heating of the metals; this is particularly the case with bismuth. The effect is attributed to the variation in crystalline structure of the metal under test at different rates of solidification. With tin the change is less marked, and with lead it is unnoticeable. At or about the melting-points there is considerable change of slope in the curves. Here, again, the effect is smallest for lead; somewhat greater with tin; and remarkably large with bismuth, the latter changing from an exceedingly active thermo-electric metal to one resembling lead. A great change occurs also with mercury at the melting-point, indicating a difference in the Peltier effect between solid and molten metals.

A vote of thanks to Mr. Beckit Burnie was proposed by the PRESIDENT, and the meeting adjourned until March 12th.

EDINBURGH UNIVERSITY CHEMICAL SOCIETY.

Fifth Ordinary Meeting, February 8th, 1897.

Dr. J. E. MACKENZIE in the Chair.

Mr. W. W. TAYLOR, M.A., B.Sc., read a paper on the "*Electrolytic Dissociation of Water.*"

The author began by describing the main principles of the theory of electrolytic dissociation.

The work of Kohlrausch in determining the actual conductivity of pure water was described in detail.

The method adopted by Wijs for arriving at the dissociation grade from the hydrolysis of methyl acetate; that of Arrhenius and others from the balance between aniline acetate and water; and, lastly, the numbers got by Ostwald and Nernst from the electromotive force of a gas battery consisting of hydrogen electrodes in acid and alkali, were all treated of and carefully explained.

Sixth Ordinary Meeting, February 22nd, 1897.

Mr. W. W. TAYLOR in the Chair.

Dr. MARSHALL gave a lecture on "*Electrolysis.*"

A historical sketch of the subject from the end of last century till the present time was first given; the invention of the electric pile by Volta, the decomposition of water by Nicholson and Carlisle, Davy's discoveries, and those of Berzelius, Faraday's laws, and the work of Daniell and Hittorf; finally, the theory proposed by Arrhenius.

The uses of electrolysis in chemical analysis were then considered, various examples being taken to illustrate this in reference to quantitative electrolysis.

A brief mention was then made of the uses of electrolysis in electrotyping, electro-metallurgy, and the purification of such metals as copper.

A selection was made both from inorganic and organic substances to show how certain syntheses could be conducted by electrolytic means.

NOTICES OF BOOKS.

A Practical Treatise on Animal and Vegetable Fats and Oils. By W. T. BRANNT. (Second Notice). Vol. II. Philadelphia: H. C. Baird and Co. London: Sampson Low, Marston, and Co., Ltd. 1896.

We find here firstly the conclusion of the account of the non-volatile or fatty oils. The efficacy of boiling as applied to linseed oil is traced to the expulsion of mucilage. It is remarkable that in some parts of Russia and Germany cold-drawn linseed oil is used as a table-oil and in cookery.

The production of sunflower oil for industrial uses in Russia is steadily increasing.

Nut-oil, expressed from the walnut, dries better even than linseed oil, and is preferred for artistic uses. Spermin oil has an advantage over most oils, as its sophistication is exceedingly difficult. The nature of ambergris is still a disputed question; some authorities considering it as a pathological secretion of the urinary bladder, whilst others regard it as merely the indurated fæces of the animal.

The remarkable penetrative power of doeglic oil was put on record as early as 1250. The credit of first obtaining cod-liver oil, colourless, not in virtue of any artificial bleaching, but of the total freedom from putrefaction, seems to belong to Peter Möller. Eulachon oil is preferred by some physicians as being more digestible than cod-liver oil.

Fish-waste is a most valuable source of artificial manure, and is capable of much extension. It is conducted chiefly in the Lofoten Isles, at Newfoundland, and on the coasts of the United States.

Borneo tallow, obtained from trees of the genus *Hopea*, is a lubricating agent far preferable even to olive oil. The cultivation of the tree deserves attention.

Cacao-butter, one of the products of *Theobroma cacao*,

and not of *Cocos mucifera* or of *Elæis guineensis*, has an exceptional power of resisting rancidity, but it is scandalously sophisticated with products which have no such properties. The original home of the cocoa-nut (better, to avoid confusion, *cocos-nut*) is correctly stated as being the Malay and the South Sea Archipelago.

It is satisfactory to find that the leading manufacturers and merchants are discouraging the term "refined lard" as applied to adulterated products.

On the subject of butters, as judged by Hehner's test, our author holds that samples with 88 per cent of insoluble fatty acids may be pronounced "pure," those with a higher percentage "suspicious," and with over 89.73 per cent "adulterated." We regret to find "titer" used in place of "standard."

Among the waxes, next to bees-wax, Chinese wax (*ceryl cerotate*) is said to possess "ten times the illuminating power of ordinary candles."

Waste fats include a variety of industrial products, one of the most important being the Yorkshire grease recovered from the waste waters of fulling mills. Its removal from the town sewage, and ultimately from the streams, is a necessary preliminary to the purification of the latter.

The bleaching of fixed oils and fats is explained very carefully. The chemical procedures were first introduced by Berthollet, though chlorine, which he proposed, has since been superseded by hydrogen peroxide, and in some cases by chromates mixed with hydrochloric acid. Animal charcoal is not well adapted for bleaching oils.

The successful application of feeble electric currents for bleaching oils seems to be due to L. Levat, of Aix. Acids could not be entirely removed in this manner from inferior lubricating oils.

Artificial butter was first produced by Mège-Mourier, under the auspices of Napoleon III. It is somewhat remarkable that whilst the Governments of France and Germany have encouraged the manufacture and sale of margarine, of course, under the proper regulations to prevent fraud, in the United States it has been opposed and persecuted in every practicable way. Eminent chemists, physiologists, and physicians, after long and careful courses of experiment, have decided that margarine has no unwholesome properties, and that it is less disposed to turn rancid than normal cow-butter.

Into the essential or volatile oils, their preparation and their distinctive characters, space does not allow us to enter.

We can strongly recommend this work to the vast and varied industries with which it is concerned.

The Chemistry of Dairying; An Outline of the Chemical and Applied Changes which takes place in Milk and in the Manufacture of Butter and Cheese; and the Rational Feeding of Dairy Stock. By HARRY SNYDER, B.S., Professor of Agricultural Chemistry, University of Minnesota, and Chemist of the Minnesota Experiment Station. Easton, Pennsylvania: Chemical Publishing Co. 1897.

THE appearance of this book is explained and justified by the change which, within the last few years, has occurred in dairying. From a mere rule-of-thumb trade to an industry which, to be successful, must be conducted on scientific principles, or, at least, in accordance with facts scientifically determined.

In the chapter on the "General Composition of Milk," the reader is reminded that milk-fat and butter are not identical. Milk-fat is the pure dry fat, free from water, casein, or salt, all of which are present in butter to the extent of about 17 per cent.

For testing milk the Babcock method is recommended as trustworthy for whole milk, but as less reliable for butter-milk and skim-milk. Milk when partially frozen is not in a condition to be sampled.

The use of pure cultures, or as they are here called

"starters," seems to be attracting attention in America for the ripening of cream.

The results of the lactometer are shown as liable to error on account of the complex composition of milk. Cream itself is variable, as it may contain as much as 60 per cent of fat. The higher the temperature reached in churning, washing, and working, the less is the proportion of water retained in the butter.

We find here no mention of the vegetable matters which in cheese-making may be used as substitutes for rennet.

It will be remarked here that in America a difference seems to be made between oleo-margarine and butterin, the former being harder and less fusible. With us this difference does not obtain, oleo-margarine being the name recognised by the law. It may here be noted that the butter now obtained from the milk of the cocoa-nut is more digestible than cow-butter.

This very useful manual is addressed not to experts, but to young men preparing themselves for the position of the dairy-farmer.

Engineering Chemistry; A Manual of Quantitative Chemical Analysis for the Use of Students, Chemists, and Engineers. By THOMAS B. STILLMAN, M.Sc., Ph.D., Professor of Analytical Chemistry in the Stevens Institute of Technology. With 154 illustrations. Easton, Pennsylvania: Chemical Publishing Company.

THE Publishing Company of Pennsylvania is decidedly earning a high and well-merited reputation for the production of works on technical chemistry. The title of the volume might with advantage be somewhat modified. Perhaps we might suggest "Manual of Quantitative Analysis, especially arranged for the Use of Engineers." But there is one section which could not be legitimately placed under such a caption. We refer, of course, to the copious and ably-written instructions for the sanitary analysis of water, a subject belonging to the medical practitioner, or the chemist truly so-called, rather than to the engineer. Here, however, we find no mention of the "tintometer" which is generally found preferable to the various calorimeters. Much of the matter on soaps can scarcely be called engineering chemistry.

On the other hand, something might have been usefully added to the remarks on filters. The Pasteur filter, which is generally found the best—at least for work on a domestic scale—does not seem to be mentioned.

The reader will be fully aware that the weights used in the United States of America are identical with the British standards. But this agreement unfortunately does not extend to the measures for liquids. The American gallon is about equal to the old English wine gallon. The obstinacy with which America, like France and Germany, clings to Baumé's hydrometer is painful. If there is any occult reason why commercial men should not or cannot use the direct specific gravity scale, why not use Twaddle, which exists only in one type, and which is easily and simply re-calculated into direct specific gravity?

Engineers will find this book a most useful and trustworthy guide.

Report on the Teaching of Chemistry by a Special Subcommittee appointed by the Technical Education Board of the London County Council, November 23rd, 1896.

WE hope that in our endeavours to realise a worthy standard of education in this country, we shall not remind the world of the too many cooks who spoiled the broth. We can scarcely enumerate the various public and semi-public bodies who have their fingers in John Bull's pie. Some boards and committees concerned are, if we consider their origin, little likely to play a very salutary part. But some of the gentlemen whose opinions

are here put on record are fully qualified to speak on higher education, and deserve a respectful hearing. Thus, Mr. D. Howard, Dr. Armstrong, and Dr. Tilden do not consider that examination selects the best students. "The best thing to be done in London to promote this higher instruction is to extend the system of senior scholarships." These scholarships should on no account be awarded as the results of an examination, but as the personal recommendation of the professors upon whom the responsibility must be thrown of nominating only students of promise. Such scholars after about two years of special training in research should be able to work by themselves." "A person who is studying for the B.Sc. degree is handicapped as far as practical work is concerned, as he has to cram for his examinations. The boys cannot be picked out by mere examination, but by careful observation of their work. Scholarships gained by examination have done higher education a good deal of injury."

In short, a careful survey of the opinions expressed by the authorities here quoted shows more than ever that the blind reliance on examinations as a test of merit is nearing its end. "The selection should rather be determined by the recommendation of the head master of the school from which a pupil may proceed, based on the work of the candidate throughout his whole school career."

This is substantially the system which has worked so successfully in Germany.

The conclusions drawn from the evidence submitted are:—

1. That chemistry is a valuable subject for school teaching.
2. That it should be preceded by elementary courses of physics.
3. That the work should be always largely practical.
4. That no attempt should be made to impart in schools any knowledge of the application of chemistry for commercial purposes.
5. That in selecting candidates for the higher science training a written examination is insufficient and inadvisable.

This report is worth the most careful attention of all who are or aim at becoming authorities on national instruction.

Selection of Procedures for the Analysis of Fuels, Iron Ores, Castings, Steels, and Irons. (Recueil de Procédés de Dosage pour l'Analyse des Combustibles, des Minerais de Fer, des Fontes, des Aciers, et des Fers). By G. ARTH, (Professeur de Chimie Industrielle à la Faculté des Sciences de Nancy. Paris: G. Carré and C. Naud. 8vo., pp. 313. 1897.

THE present development of the iron and steel manufactures, and the extension of the industrial applications, has naturally led to the appearance of such works as that before us.

The author tells us in this preface that he does not offer a treatise on analysis, but merely a selection of the procedures in use in the principal siderurgical establishments and of other methods which it may be useful to know. He remarks that industrial analyses are of two different kinds. On the one hand easy and rapid methods for daily use, and on the other difficult procedures for establishing the composition of substances taken as types, or for checking doubtful results or such as have been the subject of dispute.

No fewer than 60 pages are here devoted to the assay and analysis of fuels. The exact difference between assay and analysis is scarcely as clear as it might be desirable. "Assay" may mean either a qualitative operation, or perhaps the determination of some leading ingredient. He determines phosphorus in the ashes of the fuel by means of the molybdenum method.

He determines the calorific power of a fuel by calorimetric methods, using the bombs of Hempel, of Mahler,

and Berthelot. A whole page plate shows the installation of the bomb at the Chemical Institute of Nancy, whilst a table exemplifies the calculation.

There is also an account of the determination of calorimetric power by evaporation, and experiments in the boilers of industrial establishments. Three steam units may be employed, those of Rankine, Brix, and Hartig, equal respectively to 537, 540.6, and 552.2 cal. Then follows an account of the values of gaseous fuels; to wit, the gases of gazogens, of blast furnaces, the gaseous mixtures of the Dawson system. A chapter is also devoted to gas analysis, i.e., gases not employed as fuels but still summed up under the head assay and analyses of combustibles.

The second part is occupied with the "assay and analysis of ores," mainly, of course, those of iron. The analysis of titaniferous irons is, however, explained at some length according to the indication of Posti. It is remarked that the presence of titanic acid introduces several causes of error in determinations executed in the ordinary manner. It is also stated that oxygenated water is an excellent reagent for detecting the presence of titanic acid, as in acid solutions containing this titanic acid it produces a fine orange-yellow.

A third part of the work discusses the analysis of metallic products, iron castings, and steels. There is also the procedure of T. Parry and J. J. Morgan for the analysis of titanium, and the determination of phosphoric acid in basic slags.

Bread and Panification; Chemistry and Technology of Baking and Grinding. ("Le Pain et la Panification Chimie et Technologie de la Boulangerie et de la Meunerie"). By LÉON BOUTROUX, Professeur de Chimie Doyen de la Faculté des Sciences de Besançon. Paris: J. B. Baillière et Fils. Pp. 358. 1897.

FRANCE has long been distinguished for its eminence in the art of baking and all the collateral branches.

Hence, an author who combines the traditional skill of his country with a full insight into the results of modern Science is well worthy of our careful attention. Professor Boutroux, moreover, has not confined his studies to French authorities, but has utilised the treatises of Birnbaum and Jago, so that he is able to lay before his readers the full results of the experience of the day.

From an analysis here given of wheats as produced in different countries we find the highest rank in percentage of gluten and albumen belongs to a Polish wheat, namely, 21.5 per cent, whilst the second place falls to Egyptian wheat with 20.6 per cent. If we contrast these figures with the white provincial wheat of 9.8, we shall not conclude that temperature and latitude are main factors in the growth of superior wheats. In a dry season, however, the nitrogen is much higher than in medium and moist years.

The different parts of the grain are by no means chemically identical, and vary in consequence in their physiological activity. The "entire flour" so much valued in Britain owes its laxative properties—annoying and even dangerous to some persons if beneficial to others—to the soluble matter of its coatings.

The use of rollers in milling was first applied in Hungary about 1874, and established itself in Western Europe since 1878.

The theory of panary fermentation is thus summed up. It consists essentially in an alcoholic fermentation by means of yeast and of the sugar pre-existing in the flour.

According to Bibra's analyses of bread the composition and even the reactions differ greatly. Some white breads, e.g., those of St. Petersburg, are neutral. The rye bread of Nuremberg and of Stockholm is acid; so is also the notorious pumpernickel of Westphalia, a wholemeal bread containing a little extra bran. A still inferior bread, Keilchen, is made in some parts of Saxony and Silesia.

The frauds committed by, or ascribed to, bakers are

classified as—(1) The use of an excess of water; (2) the use of damaged flours; (3) the addition of alien flours; and lastly (4), the addition of saline waters other than sodium chloride.

This book will prove very valuable to public analysts, medical officers of health, and pharmacists.

Conspectus of Chemical Analysis. Part I.—Qualitative Analysis. ("Précis d'Analyse Chimique." Première Partie.—Analyse Qualitative). By E. FINK, Chef des Travaux Pratiques d'Analyse à l'Ecole de Physique et de Chimie Industrielle à la Ville de Paris. Paris: Georges Carres. 1896.

THIS little manual describes, in the first place, the reactions in the dry way, and then the reagents employed in the solid and in the liquid state. The author then proceeds to the assay, and in the dry way, including the use of the spectroscope. Next follows a classification of the metals from an analytical point of view, in which we perceive that not a few of the rarer substances are omitted. Next we come to the reactions of the acids, classified as the sulphuric, the hydrochloric, the nitric, the oxalic, the succinic, and acetic groups.

What may be called an Appendix treats of the preparation of the substances to be analysed, the systematic procedure for the detection of bases and acids, and the arrangement of the results.

CORRESPONDENCE

HOW SOON SHALL STUDENTS BEGIN THE STUDY OF QUALITATIVE ANALYSIS?

A REJOINDER.

To the Editor of the Chemical News.

SIR,—We perused Mr. Beebe's article on the above question with interest, but we are quite unable to agree with the conclusions he draws.

Mr. Beebe has three objections to the student beginning his laboratory course with the study of the preparation and properties of simple gases, &c. We take these objections in order.

1. "*The experiments are dangerous.*" Quite true, if the manipulation is careless; but is not a careless student just as likely in qualitative analysis to send half a test-tube of hot acid into his neighbour's face, as to blow him up with a flask of impure hydrogen when preparing that gas? And if a spirit of recklessness is abroad in the laboratory, whose fault is it? Mr. Beebe mentions hydrogen and phosphorus especially, in this connection. If the former be prepared in a small flask, and not a Woulff's bottle, the danger is lessened. All experiments, too, can be performed with small quantities of the gas; and the combustion of hydrogen, to show the production of water, is of course left till late in the lesson, when the flasks are free from air. Students should be warned of any dangerous practice.

Phosphorus need not be burned in oxygen, since carbon and sulphur could be used, but small fragments may very well be given out by the demonstrator as required, and the experiment performed.

Here, in an organised science school, we have classes of boys and girls, from eleven to fourteen years of age, working two lessons a week, and no accident whatever has occurred under the new Science Syllabus for the past two years.

But, assuming that such a course is dangerous, we fail to see that practice in warming liquids in test-tubes, or in filtering off precipitates, trains a student to handle the evolution flask or the deflagrating spoon. An hour's drill in the fitting up and correct use of apparatus would accomplish far more in this respect.

2. *Unconnectedness.* By this term Mr. Beebe would apparently have us understand that opportunities for revision are wanting in the course he condemns. His

ideal seems to be after a "This-is-the-house-that-Jack-built" principle. A series of lessons dealing with non-metallic elements can be made to follow one another in logical sequence, and when twelve have been covered it is a good plan to repeat them. But the constant repetition, such as is obtained in qualitative analysis, soon produces mechanical work, and has then little educational value.

3. *Interest.* This can certainly be well maintained by the course Mr. Beebe criticises. Variety is always interesting, and the preparation of simple gases, supplemented by easy quantitative experiments, gives far more scope in this direction than does qualitative analysis. Our young students give unmistakable proofs that they find their work here interesting.

Again, Mr. Beebe says that the student should have a good knowledge of the preparation and properties of non-metallic elements before—or at any rate while—he is engaged in qualitative analysis. Our experience is that this knowledge is very slowly gained from "lectures," no matter how ably "illustrated by experiments" they may be. What things of mystery our chemical lectures were to us as youngsters twenty years ago! But directly a student begins to do the experiments for himself, and record his own observations and inferences in his notebook, the assimilation of this knowledge proceeds apace.

Finally, is there nothing to be said as to the relative educational value of the two systems? We would train our pupils to be deft in manipulation, keen to observe and discriminate, and accurate in drawing an inference.

The use of test-tubes and reagent bottles, the colours and appearances of precipitates, do not carry us far towards such a goal, while the "inferences" are mainly matters of memory. That which by constant repetition is performed mechanically ceases to have any value as a mental exercise, and young students soon acquire a very mechanical way of "getting out" salts.

But, on the other hand, in determining the properties of a common gas, the pupil's attention is kept on the *qui vive* all the lesson; he has fresh observations to chronicle and new inferences to draw continually, while the arrangement and fitting up of his varied apparatus cultivates his manipulative powers. Nor must the quantitative side be forgotten. A lad of twelve, who has worked with us six months, with one practice lesson weekly, has just found experimentally that 145 m.grms. of mercuric oxide, when heated in a combustion-tube, give off 6.2 c.c. of oxygen, and leave a residue of mercury weighing 133 m.grms. Such an exercise has done more for his education than the qualitative analysis of half-a-dozen simple salts.—I am, &c.,

H. WIGLEY, B.A., F.C.S.

Winsford.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 5, February 1, 1897.

Constitution of the Combination of Antipyrine with the Phenols.—G. Patein.—The author infers that—1. Monomethylphenyl pyrazolone does not combine either with phenols or acid phenols. 2. Of the two atoms of nitrogen in antipyrine, the nitrogen 1, being entirely in the same relations in the mols. of dimethyl pyrazolone and of monomethyl pyrazolone, antipyrine fixes the phenols by means of nitrogen 2. 3. The existence of the combinations of antipyrine and the phenols cannot be reconciled with the supposition of E. von Meyer, according to which antipyrine might be considered as a sort of betaine.

Determination of Lipase.—MM. Hanriot and Camus.

Separation of Glycerin in Wines by Elimination in Watery Vapours.—MM. F. Bordas and Sigoda Raczkowski.

NOTES AND QUERIES.

** Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Purifying Mercury.—I shall be obliged if any correspondent can tell me how to purify a quantity of mercury I have got. Some zinc has been dissolved in it. I have no apparatus for distilling the mercury properly, so that method is impracticable. At present the mercury adheres to glass, and so is useless for a large number of experiments.—J. MacGRUBOR.

MEETINGS FOR THE WEEK.

- TUESDAY, 9th.—Royal Institution, 3. "Animal Electricity," by Prof. A. D. Waller, F.R.S.
WEDNESDAY, 10th.—Society of Arts, 8. "The Prevention of Fire Due to the Leakage of Electricity," by Frederick Bathurst.
THURSDAY, 11th.—Royal Institution, 3. "Greek History and Extant Monuments," by Prof. Percy Gardner, F.S.A.
Society of Arts, 4.30. "Prevention of Famine in India," by Sir Charles A. Elliott, K.C.S.I.
FRIDAY, 12th.—Royal Institution, 9. "The Source of Light in Flame," by Arthur Smithells, B.Sc., F.I.C.
Physical, 5. "Mechanical Cause of Homogeneity of Structure and Symmetry Geometrically investigated, with special application to Crystals and Chemical Combination," by William Barlow.
SATURDAY, 13th.—Royal Institution, 3. "Electricity and Electrical Vibrations," by Right Hon. Lord Rayleigh, M.A., F.R.S.

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1946.

VOLUMETRIC ESTIMATION OF SULPHUR IN IRON, STEEL, AND SULPHIDES OF IRON.

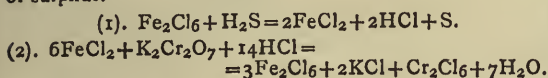
By G. G. BOUCHER.

THERE is only one volumetric process, I believe, for the estimation of sulphur in iron and steel in general use, viz., the iodine method. It is fairly accurate and quick, but has one great disadvantage—the solutions do not keep well. If kept for any length of time they lose their strength, especially if exposed to sunlight, and consequently have to be frequently re-standardised. It occurred to me that if it were possible to obtain a process where only one solution is used, and that one a solution which would keep its strength for any length of time, it would be a distinct advantage.

This I found could easily be done; and the following process will, with care, be found to give very good results, which will compare well with those given by the iodine method.

The apparatus used in this method is the same as that used in the iodine process.

From 10 to 15, or even 20, grms. of iron or steel are weighed out into a flask, which is then connected to the U-tube. Fifteen c.c. of a solution (strength 5E) of NaHO are run into the U-tube, and then sufficient HCl (strength 5E) run into the flask to dissolve the iron. The flask and contents are gently heated, and when the metal has completely dissolved the solution is heated to boiling. The U-tube is now disconnected, and the contents run slowly into an acid solution of Fe_2Cl_6 . H_2S is liberated, and a portion of the iron is reduced. The reduced iron is then estimated by a standard solution of $\text{K}_2\text{Cr}_2\text{O}_7$, and it will be evident from the formulæ below that each c.c. of $\text{K}_2\text{Cr}_2\text{O}_7$ used will be equivalent to a certain quantity of sulphur.



By dissolving 3.065 grms. of $\text{K}_2\text{Cr}_2\text{O}_7$ in 1000 c.c. of distilled water, a solution is obtained 1 c.c. of which is equivalent to 0.0008 grm. sulphur. A solution of twice this strength is used for the estimation of small quantities, but when the sulphur is believed to be above 0.15 per cent, it is advisable to use a stronger solution, and the standard solution used in the analysis of iron ores is used, 1 c.c. = 0.00571 grm. of sulphur.

The ferric chloride solution used is made by adding a solution of ferric chloride, containing 0.1 grm. of iron in 1 c.c., to 50 c.c. of hot HCl (strength 5E). The amount of ferric chloride required should be calculated, as it is not advisable to have a large excess; about 0.5 or 1 c.c. is usually found sufficient.

The bichromate solution can be standardised by pure iron wire or ferrous ammonium sulphate, but a better method is to dissolve about 15 grms. of iron or steel, in which the sulphur has already been determined gravimetrically, and treat the metal as described above. The amount of bichromate required to oxidise the reduced iron, divided by the amount of sulphur known to be present in the 15 grms. weighed out will give the value of each c.c. in sulphur.

Sulphur in Sulphides of Iron.

If the sulphide is soluble in HCl, 0.5 grm. is weighed out into a 300 c.c. flask, dissolved in about 40 c.c. HCl (strength 5E), and treated in the same way as iron. If

the sulphide is insoluble in HCl, 0.5 grm. is weighed out into a porcelain crucible and mixed with an excess of pure iron (6 grms.). The crucible is then filled up with ground charcoal, covered with a lid, and heated to redness in a muffle with a closed door for ten or fifteen minutes. The crucible is then taken out, allowed to cool, the contents emptied into a 300 c.c. flask and treated as soluble sulphide.

It is advisable to make a blank experiment, using the same quantity of iron, &c., required by this method. The iron is sure to contain a little sulphur, which would make the results appear higher than they should be. Below I give some analyses by this method.

Bessemer Iron.

	Gravimetric method. S per cent.	$\text{K}_2\text{Cr}_2\text{O}_7$ method. S per cent.
Sample 1	0.041	0.038
" 2	0.015	0.017
" 3	0.020	0.023
" 4	0.028	0.030

Mottled Iron.

Sample 1	0.312	0.320
" 2	0.411	0.399
" 3	0.295	0.296
" 4	0.384	0.387

Sulphides of Iron (Insoluble).

Sample 1	48.61	49.25
" 2	30.68	30.97
" 3	32.56	32.14
" 4	51.0	51.10
" 5	0.707	0.773

The process is a quick one; an analysis of iron, steel, or sulphide for sulphur can be made comfortably in about half or three-quarters of an hour. When the percentage of sulphur is very low considerable care is required at the finish of the process; the indicator should show no sign of any blue colour for the space of one minute.

I tried this method on a sample of copper sulphide with the object of seeing whether it would be possible to determine the sulphur volumetrically. I expected that some of the sulphur would be detained by the copper, but thought the experiment worth trying. The copper sulphide was first heated with pure iron in a muffle, and afterwards treated just as sulphide of iron. The results I give below:—

Copper Sulphide.

	Gravimetric process. S per cent.	Volumetric process. S per cent.
Experiment 1	24.0	22.30
" 2	24.0	22.74

The idea of heating the iron sulphide with pure iron in a muffle is not mine; it is a process devised by F. P. Treadwell, and can be found in the *Journal of the Chemical Society*, vol. ix.

Laboratory, North Lonsdale Iron Works,
Ulverston.

ESTIMATION OF BORIC ACID IN FOODS.

By L. DE KONINGH.

THE estimation of boric acid, at one time very troublesome, has become quite an easy matter since the discovery that it may be accurately titrated in presence of glycerol with phenolphthalein as indicator. To apply the process to articles of food (and I will confine myself for the present to milk and uncooked eggs) some few precautions must, however, be taken.

Boric acid is seldom used alone, but mostly in admixture

with borax, a mixture of three parts of the acid with one part of ground borax, constituting the article known as *glacialine*. In practice it is, however, in my opinion, not necessary to make a distinction between the acid and its sodium salt, as both are no doubt equally harmless in small quantities; but if the amount of either reaches 1 per cent or more the time has come for a protest.

When testing uncooked eggs (the entire contents beaten up) for boric acid, I take 5 grms. of the sample, add one drop of sodium hydroxide (1 : 1), dry, and finally incinerate. The char is then powdered, boiled with water, and the residual black mass again burnt. The ash is also boiled with water, and the two solutions are united. The liquid is now faintly coloured by methyl orange, and tenth-normal sulphuric acid is added until a faint pink is obtained. The solution is now boiled for a minute to expel carbon dioxide, cooled, mixed with one-half of its bulk of glycerol, and titrated with tenth-normal sodium hydroxide, with phenolphthalein as indicator. Although it is now admitted that in presence of glycerol the amount of acid may be calculated from the number of c.c. of sodium hydroxide used, I prefer to check my sodium hydroxide with pure crystallised boric acid, using about the same quantity as present in the sample, and mixing this up with exactly the same amount of glycerol and water. Working in this manner the estimation of boric acid is, as regards accuracy, second to none.

It must, however, be remembered that eggs contain a small quantity of alkaline phosphates, and that phosphoric acid behaves somewhat like the boric acid. A method of removing this acid has already been proposed and is based on the insolubility of calcium phosphate and the comparatively large solubility of the borate. I find, however, that in uncooked eggs there is just enough phosphoric acid to account for 3 c.c. tenth-normal sodium hydroxide, when working on 5 grms. of sample, so I now propose to deduct 3 c.c. of sodium hydroxide from the number of c.c. taken by the sample. I scarcely need point out the necessity of proving the acid by the alcohol test. As a rule the presence may be ascertained by simply stirring some of the sample with a drop of sulphuric acid and a little spirits of wine and then setting fire to it.

When dealing with milk I allow 1 c.c. of sodium hydroxide for every 10 grms. of the sample. If the amount of acid is, as usual, very small, no particular accuracy can be claimed for the process; but if present in larger, and consequently harmful, quantity, the results are all that may be desired.—*Journal of the American Chemical Society*, xix., No. 1, p. 55.

ELECTRIC SHADOWS AND LUMINESCENCE.*

By Prof. SILVANUS P. THOMPSON, D.Sc., F.R.S., M.R.I.

(Continued from p. 113).

HAVING touched all too briefly upon the researches of Lenard, it remains for me to speak of those of Wiedemann, of Erlangen, who for many years has made a study both of the phenomena of electric discharge and of those of fluorescence and phosphorescence. In a research made in the year 1895 he attained some results of singular interest. He had been making electric discharges, in collaboration with Prof. Ebert, by a special apparatus for producing electric oscillations of high frequency. This apparatus, in the modified form given to it by Ebert (*Wiedemann's Annalen*, liii., p. 144, 1894), stands on the table before you. It is an apparatus of the same class as that described here some years ago by Oliver Lodge, for producing Hertzian waves. An oscillating spark is produced between two polished balls set between two condensers, A and B, each made of plates of sheet zinc

(Fig. 7) a few m.m. apart. Their external circuit is however, led into the primary of a small induction-coil, the secondary of which goes to a third condenser, c. When sparks from the Apps coil are sent to the spark-gap, the oscillations of the two primary condensers set up secondary oscillations in the third condenser, to which a

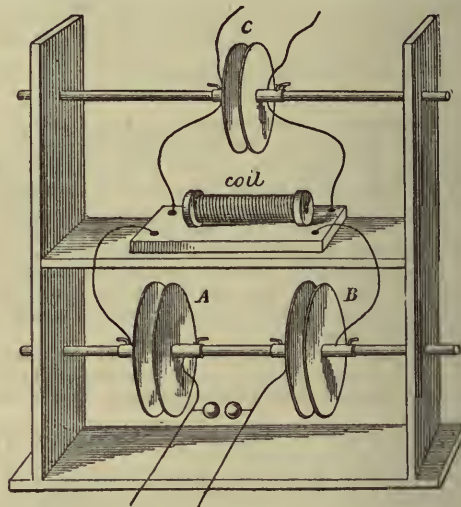


FIG. 7.

vacuum tube can be connected. If, now, by adjusting the distances between the plates of condensers, we tune the primary and secondary circuits together, the electric oscillations that result will persist much longer than if the circuits are not so tuned. Though each oscillation may

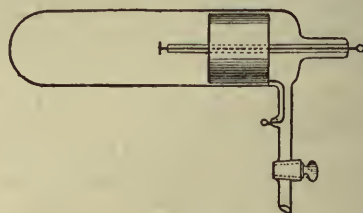


FIG. 8.

last less than the 100-millionth of a second, there will be at each spark some 20,000 or 30,000 oscillations before they have died out. Wiedemann and Ebert have found that these persistent oscillations are specially adapted to



FIG. 9.

excite luminescence. To illustrate the point I select here an old Geissler tube with a comparatively poor vacuum. When stimulated by ordinary sparks directly from the Apps coil through the platinum electrodes at its ends, it

* A Lecture delivered at the Royal Institution of Great Britain, Friday, May 8, 1896.

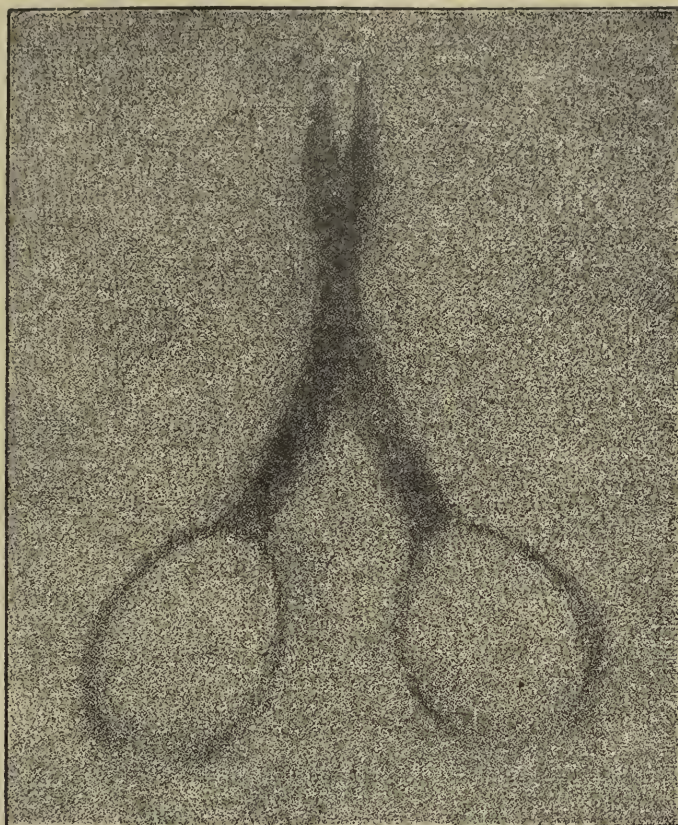


FIG. 10.

shows the usual features of Geissler tubes: there is a luminous column extending through the central bulb with stratifications along its length, while around the kathode is the usual violet glow. The glass shows no fluorescence. I now charge the connections, uniting the wires from Eber's apparatus, not to the terminal electrodes of the tube, but to two patches of tin-foil stuck upon the outside of the central bulb. Under these conditions the electric oscillations illuminate the central bulb with a glow quite different from that previously seen. Beneath each patch of foil you can discern the bluish kathode discharge, and the glass now shines with characteristic apple-green fluorescence. By moving one plate of one of the condensers in or out, I alter the conditions of resonance in

the circuit, and when the tuning is best the fluorescence is at its brightest. Now Wiedemann observed (*Zeitschr. fur Elektrochemie*, July, 1895, p. 159) that the light so generated is capable of exercising a photographic action, and of other effects, but is incapable either of passing through a thin plate of fluor-spar or of being deflected by a magnet. These rays differed, therefore, both from ultra-violet light and from kathode rays; hence Wiedemann pronounced them to consist of a new species which he named "Entladungsstrahlen," or discharge-rays. It is again a matter for research to determine whether Wiedemann's rays are the same as Lenard's, or as Röntgen's rays. Wiedemann's coadjutor Ebert went on with the research, and produced on this principle a little

TABLE I.

Kind of rays.	Cause luminescence.	Penetrate aluminium.	Cause photographic action.	Cause combination of H and Cl.	Deflected by magnet.	Discharge electrification.	Affect spark length.	Change Nature of electric discharge.	Restore thermoluminescence.	Capable of being polarised.	Capable of refraction, &c.
Ultra-violet light	Yes	—	Yes	Yes	No	If —	Yes	—	Yes	Yes	Yes
Infra-red light ..	No	No	—	No	No	No	—	—	No	Yes	Yes
Hertzian waves .	No	No	—	No	No	—	Yes	—	No	Yes	Yes
Kathode rays ..	Yes	If thin	Yes	—	Yes	(Charge violently)	—	—	—	—	—
Lenard rays ..	Yes	Yes	Yes	—	Partly	Yes	Yes	Yes	—	—	—
Wiedemann rays	Yes	Yes	Yes	—	No	—	—	—	Yes	—	—
Röntgen rays ..	Yes	Yes	Yes	—	No	Yes	Yes	Yes	Yes	No	No
Bequerel rays..	—	Yes	Yes	—	No	Yes	—	—	—	Yes	—
Electric effluve..	Yes	No	Yes	Yes	?	—	—	—	Yes	No	No

"luminescence lamp" having two external rings of foil as electrodes; and within the vacuum bulb a small pastille of phosphorescent stuff, which, when excited by the oscillations of the tuned circuits, glows with a small bright light. Ebert claims that its efficiency is many times greater than that of the ordinary glow lamp.

Returning now to Röntgen's researches, we will take a glance at the kind of tube (Fig. 8) which he was employing when he made his discovery of the X-rays. Its general resemblance to previous tubes* is self-evident. The anode was a piece of aluminium tube through which passed the glass-covered kathode wire, with a small flat aluminium plate on its extremity. From this flat plate kathode rays shot forward against the bulging end of the tube, and, without any aluminium window rays which were capable of exciting fluorescence, found their way through the glass walls. Lenard had so boxed up his tube with brass cap and metal case, that if anything in the way of rays struggled through the glass walls of his tube he might not notice it. Possibly he never looked for it. Röntgen made the fortunate observation that when his tube was closely covered with opaque black card it still could cause fluorescence on a screen covered with platino-cyanide of barium on which shadows were cast. From seeing the shadows thus to securing their imprint permanently on a photographic plate was but a small step, and the discovery that they could pass freely through a sheet of the metal aluminium was the natural result of an inquiry as to the transparency of different materials. Aluminium is to these rays much more transparent than ordinary glass. No lens can focus them, nor mirror reflect them, and, unlike the kathode rays within the tube, they are not deflected by the magnet.

The criterion which we have at present as to whether any rays from any other source are or are not the same as the X-rays is that they shall be able to fulfil the following fourfold test:—They must be capable of exciting luminescence; they must be capable of impressing an image on a photographic plate; they must be capable of passing through aluminium; and they must be incapable of being deflected by a magnet. In addition they must—so far as present evidence goes—be incapable of being either refracted or polarised. Any rays that will fulfil these tests must for the present be considered identical with X-rays.

Now it has been suggested that the X-rays are the same as ultra-violet light. This is certainly not so, for ultra-violet light, as known to us by the researches of Stokes, Tyndall, Becquerel, and Cornu, will not go through aluminium and is not deflected by a magnet, though it will excite luminescence and take photographs. Furthermore, ultra-violet light can be refracted and polarised.

It has also been suggested that the X-rays are merely invisible heat-rays. But this is certainly untrue also, because although Abney has succeeded in taking photographs by heat-rays, they will not go through aluminium, are not deflected by the magnet, and instead of exciting phosphorescence they destroy it, as Goethe found out nearly a hundred years ago.

Neither are they Hertzian waves of longer period than the heat waves.

So far as is at present known there is no other way of producing the X-rays than that of employing the highly exhausted vacuum tube. They are not found in the light of ordinary electric sparks in air. They are not discoverable amongst the rays emitted by ordinary Geissler tubes with a low exhaustion. They are not found in sunlight or any artificial light. The arc light, though it yields rays that will give photographic shadows through a thin pine-wood board, yields no rays that will pass through aluminium. The only other rays that seem to come within reasonable possibility of being X-rays are the Lenard rays, some of which are probably identical with Röntgen's; the Wiedemann rays, which are, so far as yet

investigated, entirely similar; and the Becquerel rays, to which some allusion will presently be made. It will, however, be convenient here to present a synoptic table (see Table I.) of the various kinds of rays and their respective physical properties.

One other physical property of the X-rays has been discovered since the publication of Röntgen's research. It was discovered simultaneously in Cambridge (by Prof. J. J. Thomson), in Paris, in Bologna, and in St. Petersburg, that these X-rays will cause the dielectricity of an electrified body, no matter whether it is positively or negatively charged.* That ultra-violet light can dielectricify bodies that have been negatively charged was previously known from the researches of Hertz, and of Elster and Geitel. This fresh discovery that X-rays will also discharge a positive electrification sets up a new physical test. Let me show you a simple piece of apparatus which I have found very convenient for the purpose of demonstrating this discovery. It is an aluminium-leaf electroscope (Fig. 9) entirely shielded from all external electrostatic influences by being enclosed in transparent metallic gauze. It is so well shielded that even when the cap is removed it cannot be charged in the ordinary inductive way, but must be electrified by direct conduction. The aluminium-leaves hang at the side of a fixed central plate as in Exner's electroscope. The containing vessel is of thin Bohemian glass. On exciting the instrument positively from a rod of rubbed glass, or negatively from a rod of rubbed celluloid, the leaves diverge. In either case, as soon as the X-rays are caused to shine upon the instrument, the leaves fall.

It occurred to me that by the aid of this property of dielectricification it might be possible to produce electric shadows without having resort to any photography. You are aware that if the surface or any part of the surface of a body is electrified, the fact that it is electrified can be ascertained by dusting over it mixed powders of red-lead and sulphur (or red-lead and lycopodium). With the aid of Mr. Miles Walker, who has worked with me all through this matter, I have succeeded in producing, on this plan, well defined shadows which will now be demonstrated to you. A clean sheet of ebonite, freed from all traces of previous electrification by being passed through a spirit flame, is laid on a properly prepared metal table. On it stands a small tray of thin aluminium, supported on four insulating legs. In this tray is placed the object whose shadow is to be cast,—for example, a pair of scissors or an object cut out in sheet lead. Over this again is placed a leaden cover with an opening above the tray, the leaden cover being designed to cut off electrostatic influences which might interfere. The tray is then electrified by a small influence machine, and while it is so electrified X-rays are sent downwards from a Crookes tube placed above. They pass down through the aluminium tray and carry its electrification to the ebonite sheet, which therefore becomes electrified all over except in the parts which are shielded by the scissors or other metallic object. The sheet of ebonite is then removed from the leaden enclosure, the aluminium tray lifted off, and the mixed powders are dusted over, adhering to the surface of the ebonite and revealing the otherwise invisible electric shadow. Fig. 10 is a shadow taken in this way. It is but right to mention that Prof. Righi, of Bologna, has independently obtained electric dust shadows in a very similar way since these experiments of mine were begun.

(To be continued).

Ammoniacal Silver Chlorides.—R. Jary.—In the case of ammoniacal chlorides the dissociation in a space occupied by water ensues in the same manner as in a vacuum.—*Comptes Rendus*, cxxiv., No. 6.

* It is of great interest to note that this identical property had been observed by Lenard a year previously as an effect of his rays. He found they would discharge an electroscope enclosed in a metal chamber, with an aluminium sheet in front, whether positively or negatively charged, and at a distance of 30 centimetres from his tube.

* It is, in fact, identical with the form described by Hertz in 1883. See *Wiedemann's Annalen*, xix., p. 810.

VOLUMETRIC DETERMINATION OF MOLYBDENUM AND VANADIUM.*

By CARL FRIEDHEIM.

(Concluded from p. 91).

HENCE they (*i.e.*, Gooch and Fairbanks) recommend to effect the whole operation in a current of carbon dioxide free from atmospheric air, for which, instead of the simple and convenient Bunsen apparatus, a more complicated distillatory apparatus is requisite, which, on account of its greater capacity, must give greater errors except we work with carbon dioxide. That better results cannot be obtained has been already mentioned.

A further reason which Gooch and Fairbanks advance for the necessity of their proposed alteration of my method cannot hold good. They consider it necessary that the solution to be decomposed during the operation should be evaporated down from a given initial volume to a fixed final volume, interrupting the process as soon as the latter is reached. They were led to this *modus operandi* "because it is not sufficient to indicate that the ebullition must be interrupted when the liquid has taken a clear green colour, and when a vapour of the colour of iodine ceases to pass over, for the green colour of the solution is formed very slowly, and we (Gooch and Fairbanks) were able to show iodine in the residue long after the green colour had been perfectly developed."

The latter phenomenon certainly takes place, but has nothing whatever to do with the reduction of the molybdenum tetroxide, but must be referred to the fact that if the contents of the flask are brought in contact with the air the hydriodic acid is at once oxidised. The longer such a solution is left in contact with carbon disulphide with access of air the more intensely it is coloured, which does not ensue if air is excluded.

The appearance of the green tinge and the disappearance of the iodine colouration shows sharply the end of the reaction, as it is manifest from results already published, even for substances whose proportion of molybdenum is unknown, and gives a more trustworthy basis than boiling down the solution to a certain volume, which is suitable only for the weights used by Gooch and Fairbanks, and cannot be at once transferred to substances whose proportion of molybdenum is unknown.

In the same connection we must notice another proposal made by the same authors. It is well known that vanadium pentoxide can be quantitatively reduced to tetroxide by potassium bromide and hydrochloric acid, and that an excellent volumetric method for determining vanadium can be secured (Holverschmidt's method) by receiving the liberated halogen in potassium iodide, and titrating the iodine set at liberty. In concert with Euler I have showed that on boiling vanadium pentoxide with potassium iodide and hydrochloric acid, the reduction can likewise be carried almost quantitatively as far as vanadium tetroxide, whilst vanadium tetroxide is formed quantitatively on the addition of syrupy phosphoric acid. Of course, we have not utilised this result for a volumetric investigation of pure vanadates, for which there was no need, since the potassium bromide method is simpler, more convenient, and more trustworthy. Ph. E. Browning (*Zeit. Anorg. Chemie*) now proposes to modify this method of reduction so that the reduction of the vanadates is effected under certain conditions in open vessels with potassium iodate and sulphuric acid; the solution, after the iodine has been expelled by boiling is neutralised with alkaline hydroxide mixed with tartaric acid and bicarbonate, and the vanadium tetroxide existing in solution is determined with a standard solution of iodine, titrating back with arsenious acid.

As an advantage over the convenient method of distillation, it is alleged that the complicated distillatory

apparatus is superfluous. To this it may be replied that the manipulation of the compendious Bunsen apparatus is the most convenient conceivable, that the supercession of a smooth titration with thiosulphate by titration with two standard solutions, in one case with a blue liquid, does not mean any progress, and that, finally, in Browning's method, the determination of the bases (*e.g.*, lead and barium) in the residue of the distillation is rendered impossible and much more difficult.

Exactly the same objections must be urged against the modification of my method for the determination of molybdenum as proposed by Gooch and Fairbanks, which consists in decomposing the molybdate in an open method and titrating it on Browning's principle.

In a subsequent memoir (*Zeit. Anorg. Chemie*) A. Fairbanks proposes to determine the phosphorus in the yellow ammonium phospho-molybdate indirectly, the molybdenum in the compound being ascertained according to the Gooch-Fairbanks-Browning method. The authors here overlook to mention that I have already recommended the volumetric determination of the molybdenum in phospho-molybdates, and the method which I have described, and that I connected with it Hundeshagen's method for titrating the total method by alkali hydroxide, therefore determining the phosphoric acid as difference. This method is, however, of interest only for the general examination of the so-called white phospho-molybdates; for the determination of the phosphorus in the yellow compound the gravimetric method of Hundeshagen-Pemberton is far simpler and more convenient than that proposed by Fairbanks.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, January 21st, 1897.

Mr. A. G. VERNON HARCOURT, President, in the Chair.

MESSRS. Charles A. Hill, Arthur Marshall, and William H. Sodeau were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. William Arbuckle, 34, Moore Street, Cadogan Square, S.W.; Masumi Chikashigé, B.Sc., Kumamoto, Japan; Alfred Foster Cholerton, Lyndum House, Lincoln Street, Leicester; Clarence Hamilton Creasey, 78, Baggeholme Road, Lincoln; James Crowther, B.Sc., West Field, Lightcliffe, Halifax; William Alfred Davis, 108, Gordon Road, Peckham, S.E.; Ernest Goulding, 18, Mercer's Road, Holloway, N.; Charles Heppenstall, Ferrybank, Arklow, Co. Wicklow; Harold Johnson, 5, Boulevard Clovis, Bruxelles; William Robert Lang, B.Sc., 5, Crown Gardens, Glasgow; Barker North, 3, Manor Terrace, Felixstowe; Herbert Spindler Pullar, Rosebank, Perth, N.B.; William Ralston, B.Sc., 337, Cathcart Road, Glasgow; John Stewart Remington, Dromore, Milverton, Leamington; Leonard Sumner, B.Sc., Butt Hill, Prestwich, near Manchester; Andrew Turnbull, Ph.D., Daldowie, Broomhouse, near Glasgow.

The certificates of the following candidates, recommended by the Council, under Bye-law I., par. 3, were also read:—

Thomas Hannibal Aquino, Gadag, Dharwar District, India; Alfred Rutter, Broken Hill, N.S.W.; Rustomji Navroji Unwalla, Bhaunagar, Kathiawar, India.

The following is the text of the letter which has been received from Professor Stanislaw Cannizzaro, acknowledging the address presented to him by the Society on the occasion of his jubilee.

To the Council and Fellows of the Chemical Society of London.

GENTLEMEN,—I beg to offer to your Society the report of the celebration of my seventieth birthday, together

* *Berichte d. D. Chem. Gesell.*

with the volume containing some of my writings which were reprinted on that occasion, and a copy in bronze of the gold medal which was presented to me.

At the same time, I beg to convey to my colleagues the expression of my heartfelt gratitude for their very flattering address, which I have received with the greatest satisfaction.

The Chemical Society won my devotion, when, in 1862, they added my name to the restricted roll of their foreign members; and again in 1872 when they honoured me by intrusting to me the delivery of the Faraday Lecture.

A further proof of their high consideration is now offered to me in this address, in which I find my labours for Science appreciated and valued in so very high a degree by authority so competent: I feel I am indebted for this favourable estimate of my merits to the extreme kindness which my English colleagues have always shown to me, and for which I now desire to express my profound gratitude.

Great is the pleasure which this fresh manifestation of their affectionate esteem has afforded me, thus assuring me that their estimate of me has in no way lessened.

Your ever true and affectionate colleague,

STANISLAO CANNIZZARO.

December, 1896.

Of the following papers those marked * were read:—

*1. "Observations on the Properties of some Highly Purified Substances." By W. A. SHENSTONE.

1. The author has compared the behaviour of oxygen under the influence of the silent discharge of electricity when saturated with water vapour, and when carefully dried. The results show that, contrary to the statement of previous investigators, oxygen is most freely converted into ozone when wet, and that well dried oxygen yields only a very minute percentage of ozone. The results obtained also shows that the ozone in ozonised oxygen is far more stable in the presence of water vapour than in its absence. That is to say, the change by which ozone is converted into oxygen is very greatly retarded by the presence of moisture.

2. Chlorine prepared by the electrolysis of silver chloride, and also carefully purified bromine and iodine, have been dried by very thorough treatment with prepared phosphoric oxide, and then presented to the action of mercury prepared for the purpose by several distinct methods and thoroughly dried. In every case the metal and the halogen interacted instantly and rapidly.

3. Highly purified chlorine, when submitted to the silent discharge of electricity, does not undergo condensation.

4. The abnormal expansion of chlorine which has been described by several observers appears to depend upon the presence of impurities in the chlorine.

Incidentally, a new vacuum tap and other novel apparatus are described in this paper.

DISCUSSION.

The PRESIDENT, Mr. H. B. BAKER, and Professor TILDEN expressed their admiration of the skill and resource which Mr. Shenstone had brought to bear in investigating a difficult problem.

Dr. THORPE stated that he had witnessed Dr. Budde's experiments at Bonn, which were conducted with great care, and he had watched the expansion of chlorine under the influence of the violet and ultra-violet rays. The chlorine had been prepared by the oxidation of hydrochloric acid, and was believed to be pure. Some subsequent observers had confirmed Dr. Budde's results, but others had failed to do so—as was now the case with Mr. Shenstone, whose painstaking enquiry, he hoped, might be the means of finally settling the question.

Dr. THORNE stated that he had observed on a large scale the instability of ozone when in contact with an alkali.

Mr. SHENSTONE, in reply, stated that he did not doubt the correctness of Dr. Budde's observations with chlorine

prepared and purified as he had described. He considered, however, that such chlorine could not be regarded as highly purified, and that moisture was almost certainly present.

*2. "The Action of Diastase on Starch." By ARTHUR R. LING and JULIAN L. BAKER.

The authors show that maltose when heated with Fehling's solution, under the conditions prescribed by Wein, reduces 1.079 grms. of copper per grm. of sugar. The table of Wein, therefore, gives results which are 4.5 per cent too low, a result also arrived at by Brown, Morris, and Millar.

They have examined in detail the products of the limited action of diastase on starch at 70°, and have separated maltose and the following unfermentable substances, which were purified to such an extent as to free them from all extraneous matter.

Maltodextrin α , $C_{36}H_{62}O_{31}$, identical with Brown and Morris's maltodextrin, but having the properties $[\alpha]_D = 180$: $R = 32.81$.

Maltodextrin β , $C_{24}H_{42}O_{21}$, identical with Prior's "achroodextrin III.," and having the properties $[\alpha]_D = 171.6$ and $R = 43$.

A substance, $C_{12}H_{22}O_{11}$, isomeric with maltose, and obtained from the unfermentable residue of that particular fraction previously called isomaltose by Lintner. It had the constants $[\alpha]_D = 156$ and $R = 62.5$, and may consist of the simple "dextrin," $C_{12}H_{20}O_{10} + H_2O$, the existence of which the authors' previous work foreshadowed. Inasmuch as it gave a small amount of crystalline osazone, it perhaps contained maltose.

When the three substances above named are treated with an excess of diastase at 60° for a few hours, the approximate reducing powers of the products are $R = 90$; 91.5 ; 94 , respectively.

There are now ample data to conclude that starch, when hydrolysed by diastase, is converted into a series of maltodextrins of gradually decreasing molecular weight and optical rotatory power, and of increasing reducing power. These appear to have the optical and reducing properties of mixtures of the original starch and maltose.

DISCUSSION.

Dr. G. H. MORRIS regretted that Messrs. Ling and Baker had given no particulars of the substances they described beyond the constants $[\alpha]_D$ and R . He had therefore no means of judging whether the substances agreed with the maltodextrin described by Mr. H. T. Brown and himself, nor was it possible to follow the authors' line of work. The constants given for maltodextrin α agreed fairly with the law of definite relation as formulated by Mr. Brown and himself; but the maltodextrin β (Prior's achroodextrin III.) did not, and the purity of this substance is therefore doubtful. He wished to learn more about the unfermentable residue of isomaltose, which the authors appeared to regard as one of the end-products of the action of diastase on starch. He did not think it necessary to enunciate a new theory of starch conversion whilst there was still so much dispute as to facts.

Mr. LING, in reply to Mr. Chapman, said he saw no reason for assuming the presence of a "stable" dextrin among the products of starch hydrolysis; ultimately maltose was the sole product. In reply to Dr. Morris, he said that much more information would be found in the paper than it had been possible to give an account of in the brief time at his disposal. It would be seen that the formulæ of the maltodextrins could not be calculated from the percentages of apparent maltose which they yielded.

*3. "The Solution Density and Cupric-Reducing Power of Dextrose, Lævulose, and Invert-Sugar." By HORACE T. BROWNE, F.R.S., G. HARRIS MORRIS, Ph.D., and J. H. MILLAR.

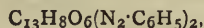
The authors have extended the methods described in

their previous paper (*Proc.*, 1896, xii., 241) to the examination of the solution density and cupric-reduction of dextrose, lævulose, and invert-sugar. They find that the solution densities of the two former differ considerably with the same concentration of the solution, but that the volume occupied in solution by a unit of weight of each is less at lower than at higher concentrations, consequently the divisor to be applied to the specific gravity decreases with the concentration. The solution density of invert-sugar was calculated from those of dextrose and lævulose, and the results so obtained were confirmed at various points by direct experiments.

They also find that the cupric-reducing powers of the three sugars, when determined under their standard conditions, are, for dextrose, $\kappa=117$ to 105; for lævulose, $\kappa=107.5$ to 101; and for invert sugar, $\kappa=111$ to 103. The higher numbers are obtained when a small amount of cuprous oxide is precipitated, and the lower when reduction is carried nearly to the maximum. When the experimental numbers are expressed in the form of a curve, it is found that at the one end, taking the cupric-reduction of dextrose at 100, lævulose is represented by 91.3, and invert-sugar by 94.2; at the other end of the curve the ratio is 100, 94.6, and 97.5; whilst at an intermediate point, which corresponds to the amount of cuprous oxide usually reduced, the relation is, dextrose 100, lævulose 92.3, and invert-sugar 96.15.

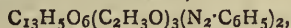
4. "Derivatives of Maclurin." Part II. By A. G. PERKIN.

From maclurin which yields a pentabenzoyl derivative (König and Kostanechi, *Ber.*, 1894, xxvii., 1996), a pentacetyl compound has not yet been obtained, for by acetylation in the ordinary way only sticky products result, and when excess of sodium acetate is employed (Ciamician and Silber, *Ber.*, 1894, xxvii., 1628), there is formed a peculiar substance having the composition of pentacetyl maclurin less 1 molecule of water. Judging from the stability of maclurin-azo-benzene,



described in a previous communication (Bedford and Perkin, *Trans.*, 1895, lxvii., 933), when compared with that of maclurin itself, it appeared probable that this on acetylation might behave normally, which was found to be the case.

Triacetyl-maclurin-azobenzene,



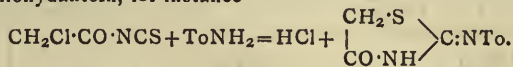
orange-yellow needles, m.p. 240–243°, is insoluble in cold alkaline solutions, but decomposed by them on boiling. Suspended in acetic acid and treated with sulphuric acid, a quantitative yield of maclurin-azobenzene is produced. Phloroglucin-azobene similarly yields a monacetyl derivative, $C_6H_3O_3(C_2H_3O)(N_2C_6H_5)_2$, orange-red needles, m.p. 222–223°, also quantitatively decomposed by sulphuric acid into the azo-compound. These substances furnished no higher acetyl derivatives, two hydroxyls present in the original molecules of maclurin and phloroglucinol having assumed in their diazobenzene compounds the ketonic condition. This method is being applied for the estimation of hydroxyl groups in certain analogous substances, particularly catechin and cyano-maclurin, which combine readily with diazobenzene, but do not give normal products on acetylation by the usual method.

Mention is made of a second product closely resembling luteolin trimethyl ether, and formed at the same time, during the methylation of luteolin. This, though isolated many months since, was not mentioned at the time, believing that the work then published was sufficient to establish priority for the further study of this reaction. The author wishes to reserve this to himself for further examination.

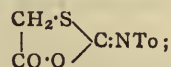
5. "Halogen-substituted Acidic Thiocarbimides and their Derivatives; a Contribution to the Chemistry of the Thiohydantoins." By AUGUSTUS EDWARD DIXON, M.D.

Continuing his previous work on the acetylthiocarbimides (*Trans.*, 1895, lxvii., 1040; 1896, lxix., 855; *ibid.*, 1593, &c.), the author endeavoured to prepare halogen substitution derivatives of certain members of the fatty acid class, in the hope that, by combination with organic bases, glycolylthioureas would be obtained of known structure, whose relations to the thiohydantoins produced by other methods would serve to decide the constitution of the latter.

The derivatives in question were obtained by heating a mixture of sand and lead thiocyanate with the α -halogenised acid chloride (or bromide), dissolved in anhydrous toluene; as a rule, the yield amounted to only about 60 per cent of the theoretical. On bringing the products into contact with primary or (secondary) amines, interaction occurred spontaneously, with elimination of the halogen, and formation of the corresponding substituted thiohydantoins, for instance—

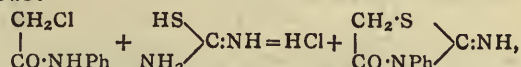


By prolonged boiling with hydrochloric acid, the latter compound is hydrolysed, ammonia being formed, together with a substance (melting at 119–120°) identical with the "orthotolylthiocarbimidoglycolide" obtained by Völtkow (*Ber.*, 1880, xiii., 1580) from $EtOH, ToNCS$ and $CH_2Cl \cdot COOH$. Since the nitrogenised organic group, introduced by the base in the formation of the thiohydantoins, does not form an integral portion of its ring, whilst, on the other hand, the nitrogen withdrawn by hydrolysis holds no organic radicle in combination, it follows that the ring must exchange its NH for oxygen, thereby becoming—

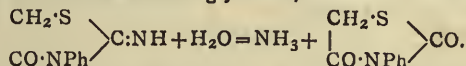


thus, by an entirely different method, the formula is corroborated, which Evers assigns to the corresponding phenyl derivative (*Ber.*, 1888, xxi., 975). From orthotolylthiourea and ethylic chloracetate, a thiohydantoins was obtained, agreeing in properties with that produced from the thiocarbimide; on hydrolysis, it afforded the same glycolide, m. p. 119–120°.

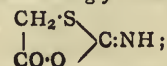
On the other hand, thiourea, when treated with a substituted chloracetamide (e.g., chloracetanilide) yielded (P. Meyer, *Ber.*, 1877, x., 1965) thiohydantoins, together with a substitution derivative; the phenylthiohydantoins so obtained was apparently identical with that produced from phenylthiourea and ethylic chloracetate. The essential interaction he explained substantially as follows:—



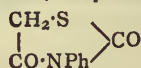
and the fact that ammonia and phenylthiocarbimidoglycolide were formed on hydrolysis, appeared to agree satisfactorily with the above view of its constitution, as well as to fix that of the glycolide,—



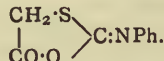
It would seem, however, from Meyer's paper, that the particular phenylthiohydantoins obtained from chloracetanilide was not used in preparing the related phenylthiocarbimidoglycolide; it was conceivable, therefore, that the former might, though melting at the same temperature, be really isomeric with that produced by the other methods. This could be ascertained by examining the products of hydrolysis, for the withdrawal from the ring of its nitrogenised group would afford aniline, together with "thiocarbimidoglycolide,"—



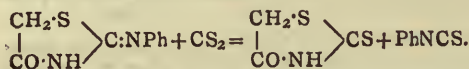
whilst, even if the phenyl group should be retained, and ammonia formed instead, the product—



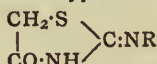
would still be an isomeride of the true phenyl thiocarbimidoglycolide,—



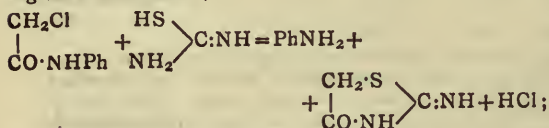
But, on experiment, the products of hydrolysis, and hence the thiohydantoïn itself, were found to be identical with those obtained in other ways. Finally, the compound was decomposed by carbon disulphide at 180°, phenylthiocarbimide being obtained, together with rhodanic acid, but not a trace of thiocyanic acid; and hence the interaction follows the course—



The author therefore regards the known monosubstituted thiohydantoïns in which the radicle is attached to nitrogen, as constituted on the type—



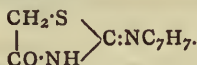
and suggests that the formation of the phenylic member from thiourea and chloracetanilide may be due to a secondary action, for thiohydantoïn is also produced, together with aniline,—



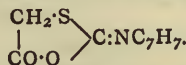
and from these, by mutual interaction, ammonia and phenylthiohydantoïn might result. Ammonia was, in fact, expelled, when aniline and thiohydantoïn were heated together with alcohol; and a substance produced, which appeared, judging from its melting-point, to be its phenylic derivative, but the quantity obtained was insufficient for analysis.

The following compounds are described:—

Orthotolylthiohydantoïn,—

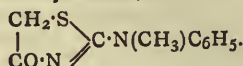


—From chloracetylthiocarbimide and orthotoluidine; white prisms, melting at 144—145° (corr.). When boiled with baryta-water it yields thioglycolic acid; by boiling, dilute hydrochloric acid, it is slowly decomposed into ammonia and "orthotolylthiocarbimidoglycolide,"—



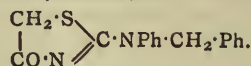
The same thiohydantoïn is produced from orthotolylthiourea and ethylic monochloracetate; its *hydrochloride* melts at 212.5—213.5° (uncorr.).

Methylphenylthiohydantoïn,—



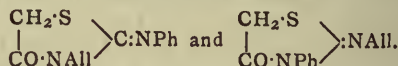
—From the thiocarbimide and methylaniline; flattened needles, melting at 129—130° (corr.), and decomposed by boiling with caustic alkali or baryta-water, into ammonia, methylaniline, and thioglycolic acid. It is also obtained by heating α -methylphenylthiourea, in alcohol, with ethylic monochloracetate; the *hydrochloride* melts at 193—194°.

Benzylphenylthiohydantoïn,—

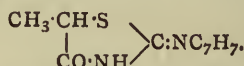


—From benzylaniline; it melts at 118—119°, and is decomposed by prolonged boiling with hydrochloric acid, into benzylaniline, and a substance melting at about 123—124°, probably "thiocarbimidoglycolide."

Allylphenylthiohydantoïn.—From allylphenylthiocarbamide and monochloracetamide an oil was obtained; it appears to be a mixture of the two forms—

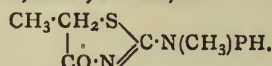


α -Bromopropionylthiocarbimide, $\text{CH}_3\cdot\text{CHBr}\cdot\text{CO}\cdot\text{NCS}$, when treated with orthotoluidine, affords *methylorthotolylthiohydantoïn*,—



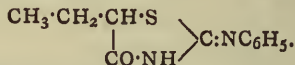
Crystalline powder, melting at 72—73°, and decomposed by boiling dilute alkali, with formation of α -thiolactic acid, $\text{CH}_3\cdot\text{CH}(\text{SH})\cdot\text{CO}_2\text{H}$.

Dimethylphenylthiohydantoïn,—



—From the above thiocarbimide and methylaniline; vitreous plates, melting at 129—130°.

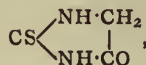
α -Bromobutylthiocarbimide, $\text{CH}_3\cdot\text{CH}_2\cdot\text{CHBr}\cdot\text{CO}\cdot\text{NCS}$, by combination with aniline, yielded *ethylphenylthiohydantoïn*,—



White needles, m. p. 148—149° (corr.).

Ethylorthotolylthiohydantoïn.—A sandy white powder, melting at 95—96°, to a turbid liquid, clearing at 98°; the *hydrochloride* forms white needles, m. p. 224—225° (corr.). When boiled with alkali, then acidified and mixed with ferric chloride, followed by ammonia, a purplish colouration is produced, due, probably, to the presence of α -thiobutyric acid.

For greater convenience and precision in naming "thiohydantoïns" of the above types, and the corresponding derivatives of—



together with the related "thiohydantoïc" acids, the author proposes a modification of the nomenclature at present employed.

6. "The Amyl (Secondary butyl-methyl) Derivatives of Glyceric, Diacetyl-glyceric, and Dibenzoyl-glyceric Acids, Active and Inactive." By PERCY FRANKLAND, Ph.D., B.Sc., F.R.S., and THOMAS SLATER PRICE, B.Sc.

The authors describe the preparation and properties of amyl (laevo-active) glycerate (dextro-active), amyl (inactive) glycerate (dextro-active), amyl (laevo-active) glycerate (inactive), as well as of the corresponding diacetyl and dibenzoyl-glycerates. The interest attaching to these bodies depends, firstly, on those compounds with the inactive amyl and active acid radicle filling gaps in the series of active glycerates, diacetyl-glycerates, and dibenzoyl-glycerates already prepared, and described by one of the authors. The position of the maximum rotation in these series becomes thus more precisely localised. Secondly, the influence of one asymmetric carbon atom on another in the same molecule can be ascertained, and the principle of the superposition of the optical effects of the asymmetric carbon atoms is put to the test and found

to hold good. Thus the authors show how the optical properties of the eight possible active amylglycerates can be calculated from a knowledge of the optical properties of two particular ones, and similarly in the case of the eight active amyl diacetylgylycerates, and the eight active amyl dibenzoylgylycerates.

In the series of the dibenzoylgylycerates, of which now the methyl, ethyl, propyl, and amyl terms are known, the rotation diminishes from the methyl to the amyl compound, and there is every probability that in this series the rotation will be found to pass through a minimum.

The influence of temperature on the rotation of all the compounds described has been also investigated, with the result that, as before, the rotation of the glycerates was found to be but little sensitive to temperature, the rotation of the diacetylgylycerates much more sensitive, and that of the dibenzoylgylycerates still more sensitive to temperature. Again, as before, it was found that the negative rotation of the diacetylgylycerates increased, whilst the positive rotation of the dibenzoylgylycerates diminished with rise of temperature. It was, however, further found that the compounds in which the amyl alone was active, viz., amyl (lævo-active) glycerate (inactive), amyl (lævo-active) diacetylgylycerate (inactive), and amyl (lævo-active) dibenzoylgylycerate (inactive), had their rotation practically unaffected by temperature, the sensitiveness to temperature being thus confined to the rotation dependent on the asymmetric carbon atom belonging to the glyceric acid part of the molecule.

7. "The Refraction Constants of Crystallised Salts." By ALFRED E. TUTTON.

This communication is in reply to certain criticisms of Pope (*Trans.*, 1896, lxi., 1530) concerning the author's work on the refraction constants of the sulphates and double sulphates containing potassium, rubidium, and cæsium (*Trans.*, 1896, lxi., 502). It is first shown that the claim of Pope to originality, in showing that the molecular refractions of solid salts are the sums of the atomic or equivalent refractions of the components, is unfounded, and that the whole of the conclusions published in the author's memoir, with regard to this subject, in connection with the entire twenty-two double sulphates investigated, were based upon the assumption of this rule. The second point is with regard to the criticism that the mean molecular refractions of the salts given were not the mean of the three values corresponding to all three refractive indices of the biaxial crystals in each case, but the mean of the two extreme values; and with regard to the re-calculated results presented by Pope taking the intermediate value into account, which Pope appears to show exhibit far greater accordance than the author's values. The author points out that the course pursued was taken after careful consideration, with full knowledge of the problem, and for the sufficient reason that the whole of the salts in question were so extremely feebly doubly refractive, and the extreme values consequently so close together, that he judged that the difference between the results of the two processes would be within the range of experimental error. He then shows that grave errors occur in Pope's re-calculations; there are numerous errors in Table III., two of them being whole numbers, one of which amounts to a fifth of the total value, and Table IV., is entirely wrong in consequence. When the errors are corrected, the latter table, in which the two whole-number errors also appear, assumes quite a different aspect, the results of the two modes of calculation become nearly identical, the differences between them being then well within the range of the experimental error, and amply justify the author's course. The author finally shows that the two cases, rubidium sulphate and cæsium sulphate, quoted by Pope as adverse to the author's statement that "the matter in a crystal has, for refraction purposes, the same average effect as the same matter uncrystallised," lead to diametrically opposite conclusions; and, moreover, that such conclusions are of no

value, as the differences in question between the values for solution and for the crystallised condition are well within the range of experimental error.

8. "The Refraction Constants of Crystalline Salts." A Correction. By WILLIAM JACKSON POPE.

The author regrets to find, notwithstanding that the numbers used in his paper (*Trans.*, 1896, lxi., 1530) were several times checked, an error of a unit in two numbers in Table III. which vitiates the first line of Table IV. (*loc. cit.*, p. 1537); the first, third, seventh, and ninth numbers in the line in question should be 5.11, 5.26, 14.5, and 15.00, and in the fourth column of Table III. the numbers 4.25 and 13.51 should each be increased by unity. The comparison made in the six lines following Table IV. is consequently unjustifiable.

The error, although to be regretted, in no way affects the general argument, but, if left uncorrected, tells unfairly against the method of calculation used by Tutton.

9. "On the Wide Dissemination of some of the Rarer Elements and the Mode of their Association in Common Ores and Minerals." By W. N. HARTLEY, F.R.S., and HUGH RAMAGE.

By means of spectrographic analysis the authors have examined about 170 specimens of ores and minerals, comprising oxides, carbonates, and sulphides. Half a grm. of each substance, finely powdered, was heated in the oxyhydrogen flame. The following elements and their compounds yield spectra under these conditions which are easily observed.

- (a) In very small quantity—Na, Ca, Pb, Ni, K, Se, Bi, Cu, Ba, Cr, Rb, Ga, Mn, Ag, In, Fe, Cs, Ti, Co.
- (b) In small quantity—Li, Au, Cd, Sb, and Sn.
- (c) In such quantity as to indicate that the substance is a principal constituent of the mineral—Be, B, Di, Te, Rh ?, Mg, Al, S, Pd ?, Zn, Ce, Se, Ru ?.

Some of the metallic elements in the list (c) under special conditions yield oxyhydrogen flame spectra, which are easily observed even in small quantity. Other elements than the above have not been sought for in this research.

In almost every case the locality from which the specimens of ores and minerals came is recorded, and the results of the spectrographic analysis have been tabulated. Several novel and interesting facts are disclosed, which may be stated very briefly as follows:—

Clay Iron-stones and Black-band Ores.—Fifty-one specimens examined. All contain sodium, potassium, copper, calcium, and manganese; 47 contain silver; 32, lead; 21, gallium; 13, nickel; 12, chromium; 1, strontium; and 1, thallium. Probably all contain rubidium, but it is difficult to recognise owing to the multitude of iron lines. Three specimens undoubtedly contain it.

Brown Hematites.—Six specimens examined. All contain sodium, potassium, copper, calcium, and manganese; 5 contain silver; 5, lead; and 5, nickel; 3, chromium; 2, gallium; 2, thallium; and 1, indium. Probably all contain rubidium; in one it is undoubtedly present.

Limonites.—Five specimens examined. All contain sodium, potassium, silver, manganese, and apparently rubidium; 4 contain calcium; 4, lead; 3, copper; 3, nickel; 1, gallium; 1, thallium; and 1, chromium.

Red Hematites.—Eighteen specimens examined. All contain sodium and potassium; 17 contain copper; 14, manganese; 13, silver; 12, lead; 12, calcium; 3 contain gallium; 3, indium; 3, nickel; 2, chromium; 1, rubidium; and 1, thallium.

Magnetites.—Seven specimens examined. All contain sodium, potassium, copper, silver, calcium, gallium, lead, and manganese. Four appear to contain rubidium; 2, nickel; and 1 contains indium.

Siderites.—Five specimens examined. All contain sodium, potassium, copper, silver, calcium, indium, and

manganese; 3 contain lead; 1 contains rubidium; 1, gallium; 1, cobalt; 1, nickel; and 1, bismuth.

Aluminous Minerals, such as Bauxites.—Seventeen specimens. All contain sodium, potassium, copper, calcium, and iron; 16 contain gallium; 15, chromium; 13, nickel; 12, manganese; 9, silver; 3, lead; and 2, rubidium.

Manganese Ores and Minerals.—Eleven specimens examined. All contain sodium, potassium, copper, calcium, and iron; 10 contain silver; 5, rubidium, and 5, nickel; 4, gallium; 4, lead; and 4, strontium; 2, barium; 1, indium; and 1, cobalt.

Blendes—Fourteen specimens examined. All contain sodium, copper, silver, and iron; 13, potassium; 12, gallium; 12, lead; 10, silver; 10, manganese; 9, indium; 7, cadmium; 4, thallium; 2, nickel; and 1, chromium. The zinc was observed in only 8 specimens, the spectrum being hidden by other lines.

Nickel and Cobalt Ores.—Nine specimens examined. All contain sodium, potassium, copper, calcium, iron, and nickel; 6 contain cobalt; 6, lead; 4, chromium; 3, silver; 1, barium; and 1, strontium.

Tin Ores.—Five specimens examined. All contain sodium, indium, and iron; 4 contain potassium; 3, copper; 3, calcium; 3, lead; 2, silver; and 2, manganese.

Galenas.—Eight specimens. All contain sodium, potassium, copper, silver, and iron; 4 contain manganese; and 3, calcium.

Pyrites.—Thirteen specimens. All contain sodium, potassium, copper, silver, calcium, and iron; 11 contain lead; 10, manganese; 5, indium; 5, thallium; 5, nickel; and 1, gallium.

Out of 168 ores and minerals examined, gallium occurs in 68; indium in 30; and thallium in 17. Rubidium occurs probably in 70, but unquestionably in 13. All the carbonates of iron and all the tin ores, without exception, contain indium. With one single exception, all the bauxites contain gallium.

Silver, copper, calcium, potassium, and sodium are very widely disseminated through all ores and minerals.

The authors draw deductions as to the formation of beds and lodes of ore from the following facts, which they claim to have established:—First, that certain groups of ores and minerals are pervaded by small quantities of the same metals as common impurities. Secondly, the rare metals, more particularly rubidium, gallium, indium, and thallium, are associated with the same groups of minerals, and also with allied groups.

It is easy to trace the association of similarly constituted compounds to their connection with elements related to each other, as determined by the periodic system of classification. These compounds have certain properties in common, distinctive of the groups of elements and compounds to which they belong; hence in a given course of chemical changes, similar compounds are formed and thrown together by precipitation or otherwise. All the minerals mentioned have undoubtedly had an aqueous origin.

The presence of the alkali metals in all the specimens, but in variable proportions, has a special significance.

In the analysis of many different precipitates, obtained both in neutral and even strongly acid solutions, the alkali metals have been found in combination with the precipitated substance. It has long been known that manganese, aluminium, and iron in the state of hydroxides, combine with more or less of the alkalis, but in a great measure such combinations have been disregarded.

Anniversary Meeting.

The Anniversary Meeting will be held on Wednesday, March 31st, at 3 o'clock in the afternoon.

Anniversary Dinner.

It has been arranged that the Fellows of the Society and their friends shall dine together at the Criterion Restaurant on Wednesday, March 31st, at 6.30 for 7 p.m.

OBITUARY.

THE LATE GEORGES VILLE.

It is our very painful duty to put on record the death of our valued friend Georges Ville, who closed his most useful and honourable career on February 22nd. The deceased held the Chair of Vegetable Physiology in the Museum of the Jardin des Plantes. His life has been essentially devoted to a practical study of the vital conditions of plants, and especially of our food-plants.

Professor Ville pointed out the necessity of keeping cultivated lands adequately supplied with those constituents of plant-food which are most readily exhausted by the crops. As such, in addition to phosphoric acid, potassium salt, and nitrogen, he laid great weight on lime in the form of gypsum. This recommendation, we need scarcely say, though borne out by his experiments, is not ratified by British practice. To keep cattle for the production of manure—as is done by too many farmers in this country—he humorously compared to the conduct of a supposed iron manufacturer who should plant and keep up forests to supply his work with fuel. He shows that all the elements of plant-food can be obtained at less cost from mineral sources. But it is a gross misunderstanding to say that Prof. Ville denounced the use of animal manures.

He introduced in France the method of trial-plots of arable land, thus making the plant analyse the soil for itself. The process which he names sideration is simply ploughing in crops which are of no value in themselves, but which serve to enrich the soil especially by fixing atmospheric nitrogen in states suitable for the nourishment of succeeding crops.

His published works, some of which have been translated into English, are of great but unequal value. His lectures on agriculture, delivered in Belgium as well as in France, cannot fail to rouse up the agricultural mind, though they carry ideas which in England would be quite out of place. But he has done an incalculable service to France, and to the civilised world in general, by exposing the worn-out fallacy that organic matter by passing through the bodies of horses or of oxen acquires some novel and mysterious virtue which it had not before.

Ville's chief works are:—"Artificial Manures" (Longmans and Co.), and "The Perplexed Farmer, how is he to meet Alien Competition?" (Longmans and Co., 1891).

NOTICES OF BOOKS.

Directory of Paper Makers, January, 1897. London: Marchant, Singer, and Co.

THE paper industry does not meet with an amount of attention from the outside public at all commensurate with its importance.

The work before us confines itself strictly to the conventional functions of a directory without entering at all upon the prospects, the difficulties, and possible dangers of the trade. The manufacture of filter-papers occupies the attention of eight firms, but we fear we must say that none of these takes a standing equal to that of Munkell and Co., of Sweden, and Schleicher and Schüll, of Germany.

A circumstance which we much regret is that the trade is so scantily developed in Ireland. There the air is less polluted with smoke than in most parts of England and even of Scotland; the water supply is more abundant in quantity, and we think we may say superior in purity to that on our side of St. George's Channel. Ireland has, in short, the greatest natural facilities for the development of the paper manufacture.

CORRESPONDENCE.

RELATION BETWEEN ROTATION AND REDUCING POWERS OF HYDROLYSED STARCH SOLUTIONS.

To the Editor of the Chemical News.

SIR,—I have not yet read the full paper of Browne, Morris, and Millar, a notice of which appears in the CHEMICAL NEWS (lxxv., p. 42).

The fact that a constant relation exists between cupric reducing and rotatory powers in hydrolysed starch solutions was pointed out by me, in a paper read before the A.A.A.S. at the Boston meeting, in 1880, and published in full in the *Proceedings* for that year, and in the *Journal of the American Chemical Society* (vol. ii., 1880, pp. 395–402). There is also an abstract of my paper in the *Berichte der Deutschen Chemischen Gesellschaft* (xiv., 1584).

From the results of my investigations, it would be only a logical conclusion to infer that similar relations exist among hydrolytic starch products in general. In the materials examined by me, the products of hydrolysis were chiefly dextrose and dextrine. In the researches made by the authors above mentioned, maltose and dextrine were the chief hydrolytic products.

In my paper the data of the examination of a large number of samples of commercial starch glucoses are found, with formulæ for calculating the percentage of reducing sugars for varying specific gravities. The calculated reducing powers obtained by these formulæ were found to agree remarkably well with the actual reducing data secured with Fehling solution. With the improved modern optical and chemical methods it is quite certain that a formula could be constructed for the hydrolytic products of starch obtained with sulphuric acid, which would give directly, from the optical observation of the solutions, practically correct figures for reducing powers.—I am, &c.,

H. W. WILEY, Chief of Division.

United States Department of Agriculture,
Division of Chemistry, Washington, D.C.,
February 24, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 6, February 8, 1897.

M. Leidier, of Marseille, addressed to the Academy a memoir on an automatic lightning-rod for telegraphic and telephonic lines.

New Researches on the Determination of Pyrophoric Acid.—M. Berthelot and G. André.—Facts to serve towards the "History of Metaphosphoric Acid," by M. Berthelot and G. André.

Reduction of Nitrates in Arable Soil.—P. P. Dehe-
rain.—These three papers will be inserted in full.

Certain Coloured Reactions.—E. Pineura.—These colourations are obtained with β -naphthol-sulphuric acid prepared by dissolving 0.02 grm. of β -naphthol in 1 c.c. of sulphuric acid of sp. gr. 1.83; we add from 10 to 15 drops of this reagent to about 0.5 grm. of the substance to be characterised; if the latter is in solution it is gently evaporated to dryness. Tartaric acid gives at first a blue colour, which, if we continue to heat, gradually passes to a very decided green tint; if, when the matter is cold, we add 15 to 20 times its volume of water we obtain a

permanent yellowish red. Citric acid gives an intense blue, which does not turn green with prolonged application of heat. The addition of 15 to 20 times its volume of water gives a colourless solution, or one very faintly yellowish. A small quantity of tartaric acid mixed with the citric acid brings up the green tint which pure citric acid never produces; 10 to 12 per cent of tartaric acid then produces a dark greenish blue. Pure malic acid gives at once a yellowish green colour, which becomes bright yellow on prolonging the heat. The addition of water turns the colour to a bright orange. These reactions are characteristic and easy to observe; it is merely necessary to heat carefully, and to withdraw the capsule from the fire as soon as any colour is observed. If it ceases to increase in intensity, we heat afresh if requisite. Other organic substances produce similar colours, but less definite and characteristic. β -naphthol sulphuric acid also serves to distinguish the alkaline nitrites; 10 drops of this reagent, added to 0.05 grm. sodium nitrite, dissolved in a few drops of water, give rise to a very intense redness, which is not altered on the addition of water. Alkaline nitrates may be distinguished in an analogous manner by means of a solution of 0.10 grm. resorcin in 1 c.c. of sulphuric acid at 1.76. The action of this reagent upon 0.05 grm. potassium or sodium nitrate determines the appearance of a red-brown, which soon becomes a very intense violet, and which passes to orange on the addition of water.

New Method of Preparing the Primary Amines.—Marcel Delépine.—It becomes easy to have a pure primary amine, if we can combine the corresponding mineral ether with hexamethylamine.

Sanitation of the Match Trade.—M. Magitot.—The author's conclusions are:—1. The sanitation of the manufacture of matches with white phosphorus is a problem simple and easy of solution. 2. The method of sanitation consists of two orders of means based on two factors of injury, which are (a) phosphorism, (b) necrosis. 3. To phosphorism we oppose the ventilation of the work by artificial means, powerful enough to withdraw the poisonous emanations from the workers. To necrosis we oppose the principle of selection; that of recruitment and maintenance from the hands of persons entirely free from any injury of the mouth or the jaws which might furnish an opening for the chemical mischief. 4. The problem of sanitation is entirely comprised in the two terms—ventilation and selection.

Determination of Potassium Bitartrate in Wines.—Henri Gautier.—This paper will be inserted in full.

Indigenous Essence of Basil (*Ocimum basilicum*).—MM. Dupont and Guerlain.—The product which accompanies linalol in oil of basil is estragol (para-methoxyallylbenzene).

Argon and Nitrogen in the Blood.—P. Regnard and Th. Schloësing.—The authors were obliged to operate on about 10 litres of blood. The blood, on leaving the veins, could not be allowed to come in contact with the air for a single instant. Whilst adhering to facts positively observed, the authors conclude that argon exists in solution in the blood.

No. 7, February 15, 1897.

The Age of Copper in Chaldea.—M. Berthelot.—Already inserted.

A Safety Receiver adapted for containing Liquefied Gases.—J. Fournier.—This paper requires the accompanying figures.

Influence of the X-Rays on the Striking Distance of the Electric Spark.—M. Guggenheimer.—The author has succeeded in establishing that:—1. At equal distances and at equal potential differences the augmentation of the striking distance of the passive spark depends on the intensity of the X-rays encountering the micrometer. 2. At an equal difference of potential (by the micro-

meter) and at an equal intensity of the X-rays the augmentation of the striking distance of the passive spark depends on the distance of the micrometer from the emissive wall of the tube. 3. The interposition of a fluorescent screen of barium platinocyanide, of a plate of glass, or of quartz does not appreciably change the radiation.

False Equilibria of Hydrogen Selenide.—H. Péla-ron.—A mathematical paper with a diagram, not suitable for insertion.

Action of Cuprous Oxide upon Solutions of Silver Nitrate.—Paul Sabatier.—Not susceptible of useful abstraction.

On certain Derivatives of Salicylic Aldehyd.—Paul Rivals.—A thermo-chemical paper. The heat of molecular combustion is = 1589.7 cal. at constant volumes and 1590.3 cal. at a constant pressure.

NOTES AND QUERIES.

* * Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Dental Alloys.—Are there any books on the alloys used in dentistry? If so, who are the publishers?—JACK.

MEETINGS FOR THE WEEK.

MONDAY, 15th.—Society of Arts, 8. (Cantor Lectures). "Alloys," by Prof. W. Chandler Roberts Austen, F.R.S.

TUESDAY, 16th.—Royal Institution, 3. "Animal Electricity," by Prof. A. D. Waller, F.R.S.

Society of Arts, 8. "The Progress of the British Colonies of Australasia during the Sixty Years of Her Majesty's Reign," by James Bonwick.

WEDNESDAY, 17th.—Society of Arts, 8. "Music in England at the Queen's Accession," by J. Spencer Curwen.

THURSDAY, 18th.—Chemical, 8. "On the Atomic Weight of Carbon," by Alexander Scott, M.A., D.Sc. "On a New Series of Micasculphates of the Vitriol Group," by Alexander Scott, M.A., D.Sc. "The Action of Alkyl Haloids on Aldoximes and Ketoximes," by Wyndham R. Dunstan, F.R.S., and Ernest Goulding.

Royal Institution, 3. "Greek History and Extant Monuments," by Prof. Percy Gardner, F.S.A.

FRIDAY, 19th.—Royal Institution, 9. "Greek and Latin Palaeography," by Sir Edward Maunde Thompson.

SATURDAY, 20th.—Royal Institution, 3. "Electricity and Electrical Vibrations," by Right Hon. Lord Rayleigh, M.A., F.R.S.

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An Associate of the Institute of Chemistry, who has had four years' experience with Consultant Analysts and four years at Works, desires engagement. Food and Agricultural work specialities. Unexceptional references.—Address, H. I., 27, Wellington Square, London, S.W.

Analytical Chemist, A.I.C., who has had experience in well-known Laboratory and Cement Works, seeks employment in Works or Laboratory.—Address, "Works," CHEMICAL NEWS Office, 6 & 7, Creed Lane, Ludgate Hill, London, E.C.

Analytical Chemist (Ph.D.) seeks Engagement; Belgian (26); studied in Germany and Switzerland; three and a half years' experience with Agricultural Laboratory; now engaged at Aniline Colour manufactories. Thorough knowledge of English, French, German, and Dutch.—Address, "A.B.K.," care of Street and Co., 30, Cornhill, E.C.

Assistant wanted, at the end of March, in a London Analytical Laboratory. Hours, 10 to 5; Sat., 10 to 1. Annual holiday, 14 days. Applicants must be experienced in the quantitative analysis of water, food, drugs, and commercial substances generally. F.I.C. or A.I.C. preferred. Salary, paid monthly, £100 to £125 a year, according to qualifications.—Apply to A.Z., CHEMICAL NEWS Office, 6 & 7, Creed Lane, Ludgate Hill, London, E.C.

Works' Chemist, A.I.C., late with large London manufacturers, well up in Plant and Building Construction, experience in management of men, and in conduction of Technical Research work, good Commercial Analyst, seeks Appointment. Moderate Salary.—Address, "Plant," CHEMICAL NEWS Office, 6 & 7, Creed Lane, Ludgate Hill, London, E.C.

Wanted, in London Smelting Works, an Assistant Chemist. Must be well up in Leady and Cuprous material.—Write C.N. 929, Deacon's Advertising Offices, Leadenhall Street, London, E.C.

Young Chemist wanted as Assistant in large Manure Works.—Apply, by letter only, to Mr. Vincent Edwards, F.I.C., West End Laboratory, 55, Weymouth Street, W.

FOR SALE.—Complete set of "Journal of the Chemical Society," from 1848–1896. 70 vols., bound in half calf; good condition. Enquiries and offers to M.Sc., CHEMICAL NEWS Office, 6 & 7, Creed Lane, Ludgate Hill, London, E.C.

BOARD OF WORKS FOR THE POPLAR DISTRICT.

TO CHEMICAL MANUFACTURERS AND OTHERS.

NOTICE IS HEREBY GIVEN,

That the Board will meet at the Board Room, 117, High Street, Poplar, on Tuesday, the 16th day of March inst., at 6.30 o'clock in the evening precisely, to receive

TENDERS

for the Supply of

DISINFECTANTS

for one year, ending Lady Day, 1898, as follows, viz.,—

CARBOLISED PEAT POWDER, 15 PER CENT OF CARBOLIC, AT PER TON.

PERMANGANATE OF POTASH, AT PER LB.

BLACK CARBOLIC ACID, 33 PER CENT OF CARBOLIC.

CHLORO-NAPHTHOLEUM, IN BULK, 25 PER CENT OF CARBOLIC.

BURNETT'S DRAIN TESTS, AT PER GROSS.

KEMP'S DRAIN TESTS, AT PER GROSS.

The Board do not bind themselves to accept the lowest or any Tender.

Contractors will be required to pay Trade Union rates of wages, and to covenant not to sub-let any portion of the Contract. Compliance with these conditions will be rigidly enforced under a penalty of the sum of £5 for each breach.

Contractors will be required to furnish securities to the satisfaction of the Board, and to enter into bond with them for the due performance of Contract, and pay down the sum of £2 immediately upon acceptance of the Tender, towards the expenses of the contract and bond.

Tenders, upon the forms for that purpose, are to be delivered, in separate covers, duly marked and sealed, before Two o'clock in the afternoon of the 16th day of March, after which time no Tender will be received.

Further particulars and Forms of Tender can be had at the Office of the Surveyor, any day to the 15th day of March, between 9 a.m. and 4 p.m.

WM. HENRY FARNFIELD,
Clerk to the Board.

Board Offices,
117, High Street, Poplar, E.,
6th March, 1897.

THE CHEMICAL NEWS.

VOL. LXXV., No. 1947.

A NOTE ON THE ESTIMATION OF ZINC OXIDE.

By E. ASTON, B.Sc. (Lond.), and L. NEWTON,
University College, London.

FRESENIUS states that zinc oxide, when mixed with sulphur, and heated gradually to redness in a current of hydrogen gas, is quantitatively transformed into zinc sulphide.

We attempted in this way to determine the purity of zinc oxide prepared in the following manner from pure zinc, and have met with an anomaly which, we think, deserves to be placed on record. A preliminary analysis of the zinc was first made, by dissolving a weighed quantity in nitric acid, evaporating to dryness, igniting, and then weighing the zinc oxide obtained.

1.106 grms. of zinc gave 1.38075 grms. of zinc oxide. Percentage of zinc in the ZnO—*Found*, 80.29; *Calculated*, 80.24.

50 grms. of zinc, thus shown to be almost pure, were dissolved in pure nitric acid, and the solution evaporated to dryness. The residue was taken up with water, and the solution filtered. To the filtrate a few drops of ammonium sulphide were added, the liquid was allowed to stand for some hours with repeated shaking, and then filtered.

The filtrate was evaporated to dryness on a water-bath, then dried in an air-bath at 150° C., and finally ignited, first in the blowpipe, and then for three hours in a muffle furnace.

Two weighed specimens of oxide thus prepared were mixed with sulphur and gradually heated to redness in a current of hydrogen gas.

After cooling down in the gas the crucibles were weighed.

This process was repeated in the one case sixteen, in the other twenty-two times, but in neither did the quantity of zinc sulphide exceed 96 per cent of the theoretical, though it had increased generally by a small amount at each weighing.

These results might conceivably be due to impurities in the zinc oxide used, and we therefore tried another method of estimation, viz., as sulphate, in order to see whether the same result would be obtained.

A weighed quantity of the oxide was dissolved in dilute sulphuric acid in a weighed platinum crucible and evaporated to dryness; the greater part of the acid was driven off by heating in an asbestos oven to about 340° C.

The crucible was placed in a sulphur bath for about two hours, then removed and weighed. It was replaced in the bath for an equal period of time, and then re-weighed (Baubigny, *Comptes Rendus*, vol. xcvi., p. 854). The weight had not changed, and the result of the experiment was that—

0.5742 grm. of ZnO gave 1.14065 grms. of ZnSO₄; hence the percentage of zinc in the zinc oxide was 80.19 instead of 80.24, the theoretical quantity.

Our zinc oxide, therefore, was pure, and, so it would seem, that the change from oxide prepared from zinc nitrate, to sulphide by heating with sulphur in a current of hydrogen, cannot be made the basis of a quantitative method.

At this point it may be advisable to give some of the numbers experimentally obtained.

(1). 0.3555 grm. of zinc oxide gave—

1st weighing	0.3735 grm. ZnS.
6th "	0.3905 " "
10th "	0.3985 " "
11th "	0.3985 " "

At this stage ZnS and sulphur were ground together in an agate mortar, so as to make sure of an intimate mixture.

14th weighing	0.4065 grm. ZnS.
16th "	0.4070 " "

At this point the quantity of ZnS obtained was 95.59 per cent of the theoretical.

(2). In this case the zinc oxide and sulphur were in each case ground together in an agate mortar, and then introduced into the crucible.

0.4685 grm. of zinc oxide gave—

1st weighing	0.5055 grm. ZnS.
12th "	0.5275 " "
13th "	0.5280 " "
15th "	0.53275 " "
16th "	0.5325 " "
22nd "	0.5374 " "

At this point the experiment was arbitrarily stopped. The quantity of zinc sulphide obtained was 95.88 per cent of the theoretical. The compound formed appears in each case to correspond roughly to a formula 3ZnS.2ZnO. Two points may be noted about these numbers:—

1. After the first heating with sulphur, the weight of sulphide produced is more than 85 per cent of the theoretical, being in the first case 87.7 per cent and in the second 90.10 per cent; any further increase is only by very small quantities, and cannot be due to impurities in the sulphur, which left no residue on ignition.

2. Two weighings may give exactly the same number; that is to say, a constant point is reached, and yet on repeating the process an increase is obtained. Thus it is impossible to find a point at which the change from oxide to sulphide definitely stops.

It was thought advisable to see whether, under the same circumstances, zinc oxide prepared from compounds other than the nitrate would be quantitatively changed into sulphide.

Experiments were therefore tried with—

- (1) Zinc oxide prepared from zinc sulphide by ignition.
- (2) " " " carbonate "
- (3) " " " sulphate "

(1). The zinc sulphide used was prepared by dissolving some of our pure zinc oxide in acetic acid, and passing into the solution a current of sulphuretted hydrogen gas.

The precipitate was collected, washed, and then dried in a current of hydrogen gas.

That this sulphide was pure is shown by the following analyses:—

(a) 0.1582 grm. ZnS gave on ignition 0.1322 grm. ZnO.		
(b) 0.11075 " " " 0.0925 " "		
	Percentage of Zn found.	Percentage of Zn in ZnS calculated by theory.
(a)	67.052	67.010
(b)	67.017	67.010

0.11585 grm. of zinc oxide thus prepared was heated with sulphur in the usual manner, and gave 0.13860 grm. ZnS.

	Percentage of Zn found.	Percentage of Zn in ZnO calculated by theory.
	80.16	80.24

(2). Zinc Oxide prepared from Zinc Carbonate.

Some zinc oxide was dissolved in acetic acid, and precipitated with pure ammonium carbonate solution.

The precipitate was collected, washed with boiling water, and then ignited until its weight remained constant. 0.2245 grm. of zinc oxide thus prepared was mixed with sulphur and treated in the usual manner, giving 0.2685 grm. of zinc sulphide.

Percentage of Zn
found.
80.14

Percentage of Zn in ZnO
by theory.
80.24

(3). Zinc Oxide prepared from Zinc Sulphate.

The zinc oxide used in this case was prepared by igniting to a constant weight the sulphate previously obtained in the estimation of zinc oxide as sulphate. 0.140 grm. of zinc oxide gave 0.168 grm. of zinc sulphide.

Percentage of Zn
found.
80.41

Percentage of Zn in ZnO
calculated.
80.24

It must be pointed out that in this case the final point was only reached after three ignitions with sulphur. But after the first ignition the quantity of zinc sulphide formed was already 98.4 per cent, and therefore the case is hardly comparable to that of the oxide prepared from the nitrate.

Thus, zinc oxide prepared by ignition of the nitrate is not quantitatively transformed into sulphide by heating with sulphur in a current of hydrogen gas, as are specimens of oxide prepared in other ways.

University College, London,
March, 1897.

THE DETERMINATION OF TITANIC ACID.

By J. JAS. MORGAN.

IN determining titanic acid in iron ores by the usual methods, part of it is found with the insoluble residue and part in the acid solution. Arnold, in his "Steel Works Analysis," describes a method based upon the principle that, in the presence of excess of phosphate of iron, titanic acid forms an insoluble phospho-titanate of iron, and is found with the insoluble residue, and, in the absence of sufficient phosphate of iron to fix the titanium in this form, brings about the desideratum by the addition of ammonium phosphate. The method on the whole is very satisfactory, the only drawback being the method of precipitating the titanium, which is effected by boiling the largely diluted solution (measuring 1000 c.c.) until the volume is reduced to 250 c.c. This occupies considerable time, while the precipitate not unfrequently adheres so tenaciously to the beaker as to render the complete removal a very difficult matter. The writer, therefore, prefers Blair's method of precipitation, and carries out an estimation as follows (a combination of Arnold's and Blair's methods):—

To the weighed portion of the dry ore add 1 grm. of ammonium phosphate dissolved in a little water, and effect solution by digesting with hydrochloric acid, and, when solution is complete, evaporate to dryness and well bake. Dissolve the dry mass in hydrochloric acid, dilute, collect the insoluble residue (containing the whole of the titanic acid as phospho-titanate of iron) on a filter-paper, and wash with hot dilute hydrochloric acid and cold water until free from iron salts. Dry the filter-paper and contents, ignite the contents in a platinum crucible, and mix with about ten times its weight of potassium carbonate. Fuse, extract the fusion in a little hot water, filter off the insoluble, and well but carefully wash with hot water. Then dry filter and contents, place in a platinum crucible, ignite, mix with about 6 grms. of acid potassium sulphate, and fuse at a low red heat for half an hour. Extract the cold fusion in 10 c.c. of hydrochloric acid and 50 c.c. of

sulphurous acid, filter, and wash with hot water. Dilute the filtrate, add 20 grms. of sodium acetate dissolved in water, then one-sixth the volume of acetic acid, and boil for a few minutes. Allow the resulting precipitate to settle, collect on a filter, and wash with water containing acetic acid. Dry, ignite, and weigh as TiO_2 .

With pig-irons the weighed portion, after the addition of the ammonium phosphate,* is dissolved in nitric acid, sp. gr. 1.20, the solution taken to dryness, and baked. The dry mass, dissolved in hydrochloric acid, again taken to dryness, re-dissolved in hydrochloric acid, and, after diluting the solution, the silica, &c., filtered off; the remainder of the operation being then similar to the above.

A RECLAMATION.

WE have received a communication from Prof. Dr. C. Riffenbach, bearing date "Cairo, February 18, 1897." The author mentions that we insert in the *CHEMICAL NEWS* of February 12th a reprint of a paper by M. Gomberg, of Michigan, which appeared in the *Journal of the American Chemical Society*, and which touches his region.

Prof. Riffenbach informs us that he published this research by Gomberg in the *Zeitschrift für Analyt. Chemie*, 1896, p. 466, as a Supplement, and requires us to notice his research. To this end he, simultaneously with this letter, sends a special reprint of the paper.†

ELECTRIC SHADOWS AND LUMINESCENCE.‡

By Prof. SILVANUS P. THOMPSON, D.Sc., F.R.S., M.R.I.

(Concluded from p. 124).

THIS will be a convenient place to mention a new effect of X-rays which I have recently observed, and which is set down in the table. When X-rays fall upon a metal object electrified by an influence machine, they produce some curious changes in the nature of the discharge into the air. If the body is already discharging itself from some edge or corner in an *aigrette* or brush discharge (visible in darkness only) the size and form of the *aigrette* is much altered. Under some circumstances not yet investigated, the incidence of X-rays causes the *aigrette* to disappear; under, others, the X-rays provoke its appearance.

Since the publication of Röntgen's research the most notable advance that has been made has been in the direction of improving the tubes. Röntgen himself has mostly employed a pear-shaped tube with a flat circular cathode near the top, producing a beautiful fluorescence of the lower part of the tube. He carefully verified the circumstance that the X-rays originate at that portion of the glass surface which receives the impact of the cathodic discharge. They appear in fact to be generated at the place where the cathode discharge first impinges upon the surface of any solid body. It is not necessary that the substance which is to act as emitter of the X-rays should become fluorescent. On the contrary, it appears that the best radiators are substances that do not fluoresce, namely the metals. I have found zinc, magnesium, aluminium, copper, iron, and platinum to answer—the last two best.§ Mr. Porter, of University College, and Mr. Jackson, of King's College, have independently found out the merits of platinum foil, the former using

* With phosphoric irons the addition of ammonium phosphate is not necessary, unless the titanium present is considerable.

† At the present date this reprint has not reached us.—Ed. C. N.

‡ A Lecture delivered at the Royal Institution of Great Britain, Friday, May 8, 1896.

§ (The author has since found metallic uranium to surpass all other metals).

an old Crookes tube designed for showing the heating effect of the cathode discharge when concentrated by a concave cathode. On the table are some of the experimental forms (see *Philosophical Magazine*, August, 1896, p. 162) of tubes I have used. The best results are found when the cathodic discharge is directed against an interior piece of metal—preferably platinum—which I term the antikathode (*Comptes Rendus*, cxvii., p. 807), set obliquely opposite the cathode, and which serves as a radiating surface from which the X-rays are emitted in all directions. When experimenting with various forms of tube, I have spent much time in watching, by aid of a fluorescent screen, their emissive activity during the progress of exhaustion. As already mentioned, X-rays are not emitted until the stage of minimum internal resistance has been passed. As the exhaustion advances, while resistance rises and spark length increases, there is noticed, by aid of the screen, a luminosity in the bulb, which, faint at first, seems to come both from the front face of the bit of platinum that serves as antikathode, and from the back face; an oblique dark line (Fig. 11), corresponding to the plane of the antikathode, being observed in the screen to separate the two luminous regions. On slightly increasing the exhaustion the emission of X-rays from the back of the antikathode ceases, while that from the front greatly increases (Fig. 12), and is quite bright right up to the angle delimited by the plane of the antikathode. There is something mysterious, needing careful investigation, in this lateral emission of X-rays under the impact of the cathode discharge.

Of all the many forms of tube yet produced none has been found to surpass the particular pattern devised by Mr. Sydney Jackson (Fig. 13), and known as the "focus tube." It was with such a tube that I showed you at the outset the fundamental experiments of Röntgen. A concave polished cathode of aluminium concentrates the cathodic discharge upon a small oblique sheet of platinum, which, while acting as antikathode, serves at the same time as anode. Not only does the concentration of the cathodic discharge upon the metal cause it to emit X-rays much more vigorously, but it also has the effect of causing them to be emitted from a comparatively small and definite source, with the result that the shadows cast by opaque objects are darker. (Photographs were then thrown upon the screen, those taken with "focus" tubes showing remarkable definition of detail. Some of these were by Mr. J. W. Giffen; others, showing diseased bones, &c., taken by the lecturer, and some by Mr. Campbell-Swinton and by Mr. Sydney Rowland, were also projected).

The objection has been taken that in these shadow photographs it is impossible to distinguish the parts that are behind from those that are in front. In a sense that is so. But I venture to say that the objection not only can be got over, but has been got over. I cannot show the proof of my assertion upon the screen, because I cannot put upon the screen a stereoscopic view. But here in my hand is the Röntgen stereograph of a dead tame rabbit. Two views were taken, in which the X-rays were thrown in two different directions at an angle to one another. When these two views are stereoscopically combined, you observe the rabbit's body with the lungs and liver inside in their relative positions. The soft organs, which cast faint shadows almost indistinguishable amid the detail of ribs and other tissues, now detach themselves into different planes, and are recognisable distinctly. I now send up for projection in the lantern the two photographs that were taken at the beginning of my discourse, and which have in the meantime been developed.

Turning back to the phenomena of luminescence,*

permit me to draw your attention to the accompanying table of the different kinds of luminescence with which the physicist has to deal.

TABLE II.

Phenomenon.	Substance in which it occurs.
1. Chemi-luminescence ..	Phosphorus oxidising in moist air; decaying wood; decaying fish; glowworm; firefly; marine organisms, &c.
2. Photo-luminescence ..	Fluor-spar; uranium-glass; (a) <i>transient</i> = Fluorescence.
	quinine; scheelite; platinum-cyanides of various bases; eosin, and many coal-tar products.
	(b) <i>persistent</i> = Phosphorescence.
	Bologna-stone; Canton's phosphorus and other sulphides of alkaline earths; some diamonds, &c.
3. Thermo-luminescence ..	Scheelite; fluor-spar.
4. Tribo-luminescence ..	Diamonds; sugar; uranyl nitrate; pentadacylparatolyketone.
5. Electro-luminescence ..	Many rarefied gases; many (a) Effluvio-luminescence.
	of the fluorescent and phosphorescent bodies.
	(b) Kathodo-luminescence
	Rubies, glass, diamonds, many gems and minerals.
6. Crystallo-luminescence ..	Arsenious acid.
7. Lyo-luminescence ..	Sub-chlorides of alkali-metals,
8. X-luminescence ..	Platino-cyanides, scheelite, &c.

You will note the names given to discriminate from one another the various sorts of luminescence. Chemi-luminescence denotes that due to chemical action, as when phosphorus oxidises, or when the glowworm emits its cold light. Then there is the photo-luminescence of the bodies which shine when they are shone upon. There is the thermo-luminescence of the bodies which shine when heated. There is tribo-luminescence caused by certain substances when they are rubbed. There is the kathodo-luminescence of the objects placed in a Crookes tube. There is the crystallo-luminescence of certain materials when they become solid; and the lyo-luminescence of certain other materials when they are dissolved. Lastly, there is the X-luminescence set up by the X-rays.

Pausing on photo-luminescence, here is an experiment to illustrate the difference between its two varieties, phosphorescence and fluorescence. Light from an arc lamp, filtered from all rays except violet and ultra-violet, is thrown upon a disc to which rapid rotation is given by an electric motor. The disc is painted with two rings, one of sulphide of calcium, the other of tungstate of calcium. Though the light falls only on one patch, you note that the sulphide shows a continuous ring of blue light, for the emission of light persists after the stuff has passed out of the illuminating rays. The tungstate, on the other hand, shows only a short trail of light, the rest of the ring being non-luminous, since tungstate has but little persistence. The light has in fact died out before the stuff has passed a quarter of an inch from the illuminating beam. This is a sort of phosphoroscope designed to show how long different materials will emit light after they have been shone upon. Those which show only a temporary luminescence are called fluorescent, while those with persistent luminescence are called phosphorescent. For many years it has been known that some diamonds are phosphorescent. Three such are here shown,* which, after exposure of one minute to the arc light, shine in the dark like glowworms. The most highly phosphorescent material yet produced is an artificial preparation of sul-

* This very convenient term was suggested some six years ago by Wiedemann, to denote the many phenomena known variously as fluorescence or phosphorescence. It refers to all those cases in which light is produced, whether under the stimulus of electric discharge, of heat, of prior exposure to illumination, or of chemical action, and the like, in which the light is emitted at a lower temperature than that which would be necessary if it were to be emitted by means of incandescence.

* Kindly lent by Dr. J. H. Gladstone, F.R.S.

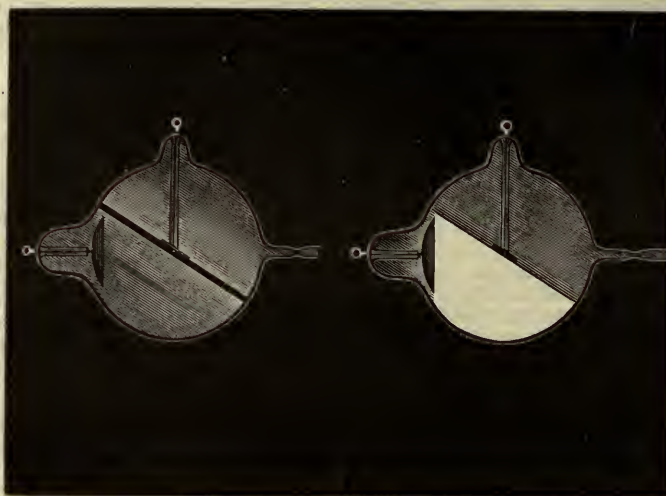


FIG. 11.

FIG. 12.



FIG. 13.



FIG. 14.

phide of calcium manufactured by Mr. Horne. The specimen exhibited has a candle-power of about $\frac{1}{10}$ candle per square inch after exposure for a few seconds to direct sunlight; but the brilliancy rapidly dies away, though there is a visible luminescence for many days. This substance is also brightly luminescent in a Crookes tube, and less brightly under the influence of X-rays.

Many substances, notably fluor-spar, have the property of thermo-luminescence, that is, they shine in the dark when warmed. Powdered fluor-spar dropped upon a hot shovel emits bright light. If, however, the spar is heated to a temperature considerably below red heat for some hours, it apparently comes to an end of its store of luminous energy, and ceases to shine. Such a specimen, even after being kept for some months, refuses to shine a second time when again heated. It has, however, long been known that the property of luminescing when warmed can be restored to the spar by passing a few electric sparks over it, or by exposing it to the silent discharge or aigrette. Wiedemann having found that the cathode rays produce a similar effect, it occurred to me to try to find out whether any of these X-rays also would revivify thermo-luminescence. I have found that on exposure for twenty minutes to X-rays, a sample of fluor-spar which had lost its thermo-luminescent property by

prolonged heating was partially though not completely revived.

I referred earlier to the rays recently discovered by M. H. Becquerel. In February last M. Becquerel, and independently I myself (see *Philosophical Magazine*, July, 1896), made the observation that uranium salts emit some rays which very closely resemble the X-rays, since they will pass through aluminium and produce photographic action. It remains to be seen whether these rays are identical with those of Röntgen.

Finally, let me briefly exhibit two results of my own work. There is now shown (Fig. 14) the photographic shadow of two half-hoop ruby rings. One of them is of real rubies, the other of imitation stones. By artificial light it is difficult to distinguish one from the other, but when viewed by the X-rays there is no mistaking the false for the true. The real rubies are highly transparent, those of glass are practically opaque.

After gaining much experience in judging by photography of the relative transparency of materials, I made a careful research (*Phil. Mag.*, August, 1896) to discover whether these rays can be polarised. At first I used tourmalines of various thicknesses and colours. More recently I have tried a number of other dichroic substances, — andalusite, sulphate of nickel, of nickel and

ammonium, sulphate of cobalt, and the like. The method used for all was the following:—A slice of the crystal was broken into three parts. One part was laid down, and upon it were superposed the other two in such a way that in one the crystallographic axis was parallel, in the other perpendicular, to the crystallographic axis in the first piece. If there were any polarisation the double thickness where crossed in structure would be more opaque than the double thickness where the structure was parallel. Not the slightest trace of polarisation could I observe in any case. Of numerous other observers who have sought to find polarisation, none has yet produced a single uncontested case of polarisation.

At the present moment interest centres around the use of luminescent screens for observing the Röntgen shadows, and in this direction some advances have been claimed of late. It should, however, not be forgotten that Röntgen's original discovery was made with a screen covered with platino-cyanide of barium. Here is a piece of card covered with patches of several different kinds of luminescent stuffs, several platino-cyanides, several sulphides, and some samples of tungstate of calcium. Of these materials the brightest in luminescence is the hydrated platino-cyanide of potassium employed by Mr. Sydney Jackson; the next brightest is a French sample of platino-cyanide of barium; platino-cyanide of strontium coming third.

Using a focus tube of Mr. Jackson's improved pattern, enclosed in a box with a cardboard front, and taking a platino-cyanide screen, I am able in conclusion to demonstrate to all those of my audience who are within a few feet of the apparatus, the facts that have so startled the world. You can see the bones of my hand and of my wrist. You can see light between the two bones of my forearm; while metal objects, keys, coins, scissors, &c., enclosed in boxes, embedded in wood blocks, or locked up in leather bags, are plainly visible to the eye.

Whatever these remarkable rays are, whether they are vortices in the ether, or longitudinal vibrations, or radiant matter that has penetrated the tube, or, lastly, whether they consist simply of ultra-violet light, their discovery affords us one more illustration of the fact that there is no finality in science. The universe around us is not only not empty, is not only not dark, but is, on the contrary, absolutely full and palpitating with light: though there be light which our eyes may never see, and sounds which our ears may never hear. But science has not yet pronounced its last word on the hearing of that which is inaudible and the seeing of that which is invisible.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, February 4th, 1897.

Mr. A. G. VERNON HARCOURT, President, in the Chair.

Mr. H. L. BOWMAN was formally admitted a Fellow of the Society.

Certificates were read for the first time in favour of Messrs. John Owen Alexander, 11, Avenue Road, South Norwood Park, S.E.; John B. Ashworth, 16, Ducie Street, Prince's Park, Liverpool; John Barclay, B.Sc., Avenue Cottage, near Bromsgrove, Worcestershire; Frank Bastow, B.Sc., 1, Braithwaite, Keighley; William Dillon, 7, Laurel Place, Chapel Lane, Armley, Lancashire; E. G. Guest, M.A., The Grammar School, Kirkham, Lancashire; T. Hartley, Gatwell Street, Bruton, Somerset; John Holmes, Crewe Villa, Putney Bridge Road, S.W.; O. C. Johnson, 52, Thayer Street, Ann Arbor, Michigan, U.S.A.; H. King, B.Sc., 4, North Street, Scarborough; H. M. Lloyd, 28, Victoria Street, Merthyr; R. N. Lyne,

Government Offices, Zanzibar; C. H. Parker, Manor House, Tettenhall, Wolverhampton; S. Pollitt, B.Sc., 19, Paulton Square, Chelsea, S.W.; M. Wildermann, Ph.D., Davy-Faraday Laboratory, Albemarle Street, W.

Of the following papers those marked * were read:—

*10. "*Observations upon the Oxidation of Nitrogen Gas.*" By Lord RAYLEIGH, F.R.S.

On the basis of Davy's assertion that the dissolved nitrogen of water is oxidised during electrolysis, various attempts were made; but they led to no useful result, even leaving it doubtful whether Davy's facts are correct.

The influence of pressure upon the oxidation of nitrogen by the electric flame was next examined. It appeared that, while in a small vessel the effect of increased pressure was favourable, but little advantage resulted when the vessel was large enough to give the maximum effect at a given pressure. The pressures compared were two atmospheres, one atmosphere, and half an atmosphere.

The remainder of the paper is devoted to a detailed description of a large scale apparatus (shown working) in which 21 litres of mixed gases enter into combination per hour, at an expenditure of about 1 horse-power.

DISCUSSION.

Professor ARMSTRONG, referring to Lord Rayleigh's remark as to the importance of the platinum electrodes being red-hot, enquired whether there was any evidence that the platinum played a special part in the process.

Professor RAMSAY suggested, as an explanation of the better results obtained when a large vessel was employed, that nitric oxide was the first product, and that this subsequently combined with oxygen to form the peroxide.

The PRESIDENT considered it probable that some of the oxide of nitrogen first formed was subsequently decomposed by the heat of the flame itself, and that the large vessel, by presenting a large surface of alkaline liquid, favoured the rapid absorption of the oxide of nitrogen, and thus less was decomposed than would be the case in a smaller vessel, where the rate of absorption was smaller. He enquired with what proportion of nitrogen to oxygen combustion occurred most rapidly.

Professor M'LEOD remarked that he had made an experiment in the manner originally suggested by Cavendish, and had found that nitrite as well as nitrate was formed.

Lord RAYLEIGH, in reply, said that the larger vessel apparently led to better results by increasing the facilities for absorption. He did not consider that the platinum played any special part in the process. The action seemed most rapid when the proportion of air to oxygen was as about 5 to 6, which corresponds with two of nitrogen to three of oxygen. He believed that both nitrite and nitrate were formed. The apparatus shown was suitable for the preliminary concentration, but not for the final purification of argon.

*11. "*On some Apparatus for Steam-distillation.*" By F. E. MATTHEWS, Ph.D.

In this paper several forms of apparatus for automatically steam-distilling substances are described.

Some solids of high melting-point may be separated by boiling the substance mixed with water in a flask fitted with a reflux condenser; the solid substance adheres to the inside of the condenser, whence it can be removed from time to time.

For liquids heavier than water, the flask in which the mixture is boiled is connected with the side tube of an ordinary distilling-flask; this disilling-flask, filled with water up to the side-tube, serves as the receiver. Into the neck of the receiver, and passing below the surface of the water, a bent Liebig's condenser is fitted which has the peculiarity of having a hole made in it a short distance above the level of the water in the receiver. On boiling the mixture in the flask, the vapours pass up the side-tube of the receiver into the upper portion of its neck and thence through the hole in the receiver, when condensation takes place. The condensed liquids run down the

condenser to the water-level in the receiver, where a drop of the heavy fluid sufficiently large to sink is formed from time to time. The condensed liquids displace their own volume of water, which flows from the receiver through the side-tube back again to the boiling-flask. In all cases in which vapour is passing in one direction in a tube, and water in the other, the advantage of perforating the tube near its lower end is pointed out.

For liquids lighter than water, the apparatus consists of the boiling-flask, which is an ordinary distilling-flask; this is connected by the upper opening to an upright tube furnished with a T-piece. The top of the upright tube is connected to the condenser, the lower end dips two or three inches into a Woulfe's bottle, containing water in sufficient quantity, which serves as the receiver. Through another neck of the Woulfe's bottle, a second tube passes from the bottom of the bottle and is connected to the side-tube of the boiling-flask. The mixed vapours pass from the boiling-flask into the upright T-tube, thence into the condenser; there becoming condensed, they fall down into the T-tube, producing a column of liquid which forces water from the bottom of the receiver back into the boiling-flask through its side-tube.

A modification of this apparatus dispenses with the necessity of having an indiarubber connection exposed to the hot vapour. In this modification the boiling-flask is an ordinary plain flask. This is connected to the condenser by a side-tube blown on to the upright tube. The water returns to the boiling-flask through another T-tube, blown on to the side-tube of the upright tube. For conveniently emptying the receiver without dismantling the apparatus, a separating funnel with two necks may replace the Woulfe's bottle of the previous apparatus.

Many liquids bump badly when boiled with water; this can generally be got over by introducing a zinc-platinum couple into the boiling-flask. The temperature of the water in the boiling-flask may be raised by dissolving suitable substances, such as sulphuric acid or calcic chloride, in it, or liquids of higher boiling-point may be used.

*12. "Researches in the Stilbene Series." I. By JOHN J. SUDBOROUGH, Ph.D.

The author has obtained benzil as one of the products of the action of zinc dust and acetic acid on benzoin; if the action is continued the benzil disappears and the chief product is deoxybenzoin. The formation of an oxidation product of benzoin by the action on it of zinc dust and acetic acid appeared so remarkable that the author has studied the action of acetic acid alone on benzoin, and he finds that small quantities of benzil are formed if benzoin is heated with six times its weight of glacial acetic acid on the water-bath for eight—nine hours.

By the action of phosphorus pentachloride on deoxybenzoin a solid chlorstilbene has been obtained, which differs from Zinin's oily compound. It melts at 45°, yields a dibromide and a dichloride, also with nitrous fumes two compounds probably represented by the formulæ $\text{Ph} \cdot \text{CHNO}_2 \cdot \text{CClNO}_2 \cdot \text{Ph}$ (m. p. 128°) and $\text{Ph} \cdot \text{C}(\text{NO}_2) \cdot \text{C}(\text{NO}_2) \cdot \text{Ph}$ (yellow prisms, m. p. 104–105°).

An oily compound can be obtained by the action of phosphorus pentachloride on deoxybenzoin at low temperatures. The oil contains the same amount of chlorine as solid chlorstilbene.

Methyldeoxybenzoin and ethyldeoxybenzoin on treatment with phosphorus pentachloride can be made to yield both oily and crystalline compounds, analysis of which points to their being methyl- and ethyl-chlorstilbenes. Solid methylchlorstilbene melts at 124°, and the corresponding ethyl compound at 60°. The dichlorides and dibromides are also described. The question as to the nature of the oily compounds has not been settled; the author describes a method by which he proposes to determine whether they are merely slightly impure forms of the solid compound, true stereo-isomerides, or structurally isomeric with the solid compounds.

*13. "Diortho-substituted Benzoic Acids. III. Hydrolysis of Substituted Benzamides." By JOHN J. SUDBOROUGH, Ph.D., PERCY G. JACKSON, and LORENZO L. LLOYD.

In order to determine whether diortho-substituted benzamides exhibit the same remarkable degree of stability towards hydrolysing agents as characterises the diortho-benzoyl chlorides (*Trans.*, 1895, lxvii., 587) and ethereal salts, the authors have investigated the following compounds:—

Ortho-, meta-, and para-brombenzamide; 2:4-, 2:6-, and 3:5-dibrombenzamide; 2:4:6- and 3:4:5-tribrombenzamide; 2:4:6-trichlorbenzamide; 2:4:6-trimethylbenzamide (mesitylformamide) and mesitylacetamide, $\text{C}_6\text{H}_2\text{Me}_3 \cdot \text{CH}_2 \cdot \text{CONH}_2$. Of the three mono-substituted brombenzamides the ortho-compound proves to be somewhat more stable in the presence of boiling (30 per cent) sulphuric acid than the meta- and para-compounds. This agrees with the properties of the corresponding methylic monobrombenzoates and of the monobrombenzoyl chlorides.

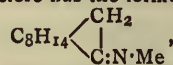
Of the three dibromamides the 2:6- or di-ortho-substituted compound proves to be the one most difficult to hydrolyse by means of 75 per cent sulphuric acid, and again of the two tribromamides the 2:4:6- or symmetrically substituted amide is much more stable than the isomeric 3:4:5-tribrombenzamide. 2:4:6-trichlorbenzamide although not hydrolysed so readily as 2:4- and 3:5-dibrom- and 3:4:5-tribrombenzamide is less stable than the corresponding 2:4:6-tribrom-compound.

The methyl derivatives could not be investigated as regards their hydrolysis with 75 per cent sulphuric acid, as they are charred and decomposed by this means. Towards 30 per cent sulphuric acid the mesitylformamide is much more stable than the corresponding acetamide.

In the course of this investigation the following new compounds have been obtained:—3:5-Dibrombenzamide, m. p. 187°; 2:4:6-tribrombenzonitrile, m. p. 127°; 2:4:6-tribrombenzamide, m. p. 193–194°; 3:4:5-tribrombenzamide, m. p. 199°; 2:4:6-trichlorbenzonitrile, m. p. 75°; 2:4:6-trichlorbenzamide, m. p. 177°; mesitylformamide, m. p. 105°; mesitylacetamide, m. p. 210°.

*14. "Conversion of Camphoroxime into Methylcamphorimine and Camphenylnitramine." By M. O. FORSTER, Ph.D.

Further investigation of the base obtained on heating camphoroxime in sealed tubes with methylic iodide has proved it to be the methyl-derivative of Tiemann's camphorimine; it therefore has the formula—



and not, as appeared probable from the preliminary examination (*Proc.*, 1895, xi., 145), the formula $\text{C}_{12}\text{H}_{10}\text{N}$. This is shown by its behaviour towards concentrated hydrochloric acid at 200°, giving rise to camphor and methylamine.

Methylcamphorimine hydrochloride and methiodide melt at 270° and 231–232° respectively; the perbromide has also been prepared.

The action of dilute nitric acid on camphoroxime, if interrupted after a few minutes, gives rise to camphenylnitramine, which is also formed when a solution of the oxime in chloroform is treated with nitrogen peroxide.

An acid solution of potassium permanganate converts camphoroxime into an unstable nitroso-derivative, which separates from the liquid as a sticky, green mass; when preserved in the desiccator the substance deliquesces, and loses its green colour, the yellow residue yielding camphor when distilled in an atmosphere of steam.

15. "Note on Wechsler's Method for the Separation of Fatty Acids." By ARTHUR W. CROSSLEY.

Wechsler (*Monatsh.*, 1893, xiv., 462) has described a method for the separation of fatty acids, the principle of which method is contained in the following statement.

If to a mixture of two fatty acids four-fifths of the caustic soda necessary to neutralise them be added, and the whole steam-distilled, the distillate contains the pure higher-boiling acid. From the residue of the distillation a further three-fifths of the acids are set free by addition of sulphuric acid, and the whole distilled in steam. Finally, the remaining fifth of the acid is set free, and in this case the distillate contains the lower-boiling acid in a pure condition.

The purity of the acids contained in the various distillates was proved by converting them into silver salts and subsequent analysis of these salts.

After trying this method of separation, with very unsatisfactory results, on a mixture of fatty acids obtained in a research on which Professor Perkin and the author have been engaged for some time past, it was thought advisable to test some of Wechsler's experimental data.

Accordingly, Wechsler's experiments have been carefully repeated and results obtained which do not agree with that author's.

As Wechsler always worked with equimolecular proportions of fatty acids, the results of some experiments are recorded in which varying proportions of fatty acids were employed. In every case the results were unsatisfactory, for even when using three molecules of the lower to one of the higher boiling acid, the former was not obtained pure in the last distillate, and the first distillate contained a decided mixture of the two acids.

It is therefore to be concluded that Wechsler's method does not give such good results as the author suggests, nor can it in any way be looked upon as a satisfactory method for separating mixtures of fatty acids.

16. "On the Crystalline Structure of Gold and Platinum Nuggets and Gold Ingots." By A. LIVERSIDGE, LL.D., F.R.S.

In view of the theory that gold nuggets are built up of concentric layers deposited round a central nucleus, the author has examined a large number of specimens from various sources. The nuggets were ground down, or sliced through, to obtain sections, which were polished and etched by suitable solvents. They all possessed a well-marked crystalline structure, and usually enclose foreign substances. The crystalline structure is not incompatible with an aqueous origin; and the author suggests that the gold has been slowly deposited from solution, either at ordinary or at high temperatures; the nuggets being more or less rolled masses of gold which have been set free from disintegrated veins.

17. "On the presence of Gold in Natural Saline Deposits and Marine Plants." By A. LIVERSIDGE, LL.D., F.R.S.

The author gives a preliminary account of the results of the examination for gold of rock salt, sylvine, and other similar saline deposits, bittern, sea-weed, kelp, oyster-shells, &c. The process of determination used was to add from 0.5 to 5 grms. of ferrous sulphate to the unfiltered solution of 100 to 1000 grms. of the salt in water. The resulting precipitate was then scorified with lead and cupelled. The natural salts contained from 1 to 2 grains of gold per ton, whilst kelp and bittern furnished in some cases as much as from 14 to 20 grains.

Ordinary Meeting, February 18th, 1897.

Mr. A. G. VERNON HARCOURT, President, in the Chair.

Mr. E. Haynes Jeffers was formally admitted a Fellow of the Society.

Certificates were read for the first time in favour of Messrs. Herbert William Leyland Barlow, M.A., M.B., Holly Bank, Urmston, Manchester; Frederick Filmer de Morgan, Andely Lodge, Caeran Park, Newport, Monmouthshire; Louis Charles Deverell, Onslow House, Worthing; William James George Lasseter, B.A., 10,

Stanley Road, Oxford; Harry Edward William Phillips, B.A., 47, Chalfont Road, Oxford; William Herbert Waite, B.A., Park Road, Halifax; Charles Thomas Foster Watts, 7, Cambrian Crescent, Chester; John Welsh, 12A, Sellar Street, Chester.

The certificate of the following candidate, recommended by the Council under Bye-law I. (3), was also read:—Frederic Hewlett Burton-Brown, Simla, India.

It was announced that the following changes in the Officers and Council were proposed by the Council:—

As *President*—Professor James Dewar, M.A., LL.D., F.R.S., *vice* Mr. A. G. Vernon Harcourt, M.A., D.C.L., LL.D., F.R.S. As *Vice-Presidents*—Professor W. Ramsay, Ph.D., F.R.S., and Professor J. Emerson Reynolds, M.A., F.R.S., *vice* Professor James Dewar, M.A., LL.D., F.R.S., and Mr. Horace T. Brown, F.R.S. As *Ordinary Members of Council*—Messrs. C. T. Heycock, M.A., F.R.S.; Rudolph Messel, Ph.D.; Tom Kirke Rose, D.Sc.; and Alexander Scott, M.A., D.Sc., *vice* Messrs. Bernard Dyer, D.Sc.; G. Harris Morris, Ph.D.; W. A. Shenstone; and T. Stevenson, M.D.

It was also announced that the Council had awarded the Longstaff Medal to Prof. William Ramsay, F.R.S., for the discovery of helium, and his share in the investigation of argon.

Messrs. H. Brereton Baker, F. D. Chattaway, and John Shields were appointed to audit the Society's accounts.

Of the following papers those marked * were read.

*18. "The Formation of Dithionic Acid in the Oxidation of Sulphurous Acid by Potassium Permanganate." By T. S. DYMOND and F. HUGHES.

When a solution of sulphurous acid is titrated with a solution of potassium permanganate, decolorisation of the permanganate ceases when only 89 per cent of the quantity required to oxidise the sulphurous acid to sulphuric acid has been used. This is due to the formation of dithionic acid in addition to sulphuric acid. The proportion of dithionic acid produced is constant, and is not influenced by either the dilution or the temperature, or the acidity of the solution. Its production, therefore, appears to be an essential part of the reaction, and to be due to the weak oxidising action of the permanganate in a final stage of its reduction. The sulphuric and dithionic acids produced are in the proportion required by the supposition that manganese heptoxide is first reduced to the red oxide with production of sulphuric acid, and further reduced to the monoxide with production of dithionic acid. When, however, sulphurous acid is treated with the red oxide, sulphuric acid is the only product.

DISCUSSION.

The PRESIDENT said that he had worked, a number of years ago, upon the reaction between solutions of potassium permanganate and sulphurous acid, before sodium thiosulphate had come into use for estimating iodine. In making determinations without excluding air from the water, he had found that the quantity of permanganate used was far less than the amount necessary for the complete oxidation of the sulphurous acid. He found that the sulphurous acid was oxidised by the atmospheric oxygen dissolved in the water, and so progressively as the water gradually dissolved the oxygen in the air lying over it. As the result of a number of experiments, he proved that the diminution in the quantity of permanganate required increased with the dilution of the sulphurous acid, and also that if the water was boiled until air-free the quantity of permanganate used was larger; but he had not obtained such constant results as Messrs. Dymond and Hughes. He had tried the experiment of adding a small quantity of manganous sulphate to the dilute solution, and had found that this salt also was able to determine the oxidation of sulphurous acid by atmospheric oxygen. He thought the author's experiments

extremely interesting in showing the constant production of dithionous acid.

Dr. SCOTT thought it would be worth while to try the effect of manganic sulphate in oxidising sulphurous acid.

Prof. DUNSTAN suggested that it would be interesting to determine whether the formation of dithionous acid occurred at the positive electrode during the electrolysis of a solution of sulphurous acid, since it seemed possible that the dithionous acid might be formed by the oxidation of sulphurous acid in much the same way as persulphuric acid was formed by the oxidation of sulphuric acid. He understood that in the remarkable action of manganous sulphate described by the President this salt undergoes no change.

(To be continued).

PHYSICAL SOCIETY.

Ordinary Meeting, March 12th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

MR. WILLIAM BARLOW read a paper on "*A Mechanical Cause of Homogeneity of Structure and Symmetry, Geometrically Investigated, with Special Application to Crystals and to Chemical Combination.*" Illustrated by Models.

The author has previously established that every homogeneous structure displays one or other of the thirty-two kinds of crystalline symmetry. He now shows that homogeneous structures possessing most, if not all, of these kinds of symmetry may be produced mechanically, as the equilibrium arrangements of assemblages of mutually-repellent particles; and, also, that these mechanical systems of particles exhibit characteristics entirely analogous to certain crystalline and other properties of matter. The fundamental concept may be summarised thus:—A number of different kinds of mutually-repellent particles dispersed through space, the amount of this repulsion being some inverse function of the distance between the particles concerned; the particles are destitute of polarity, and the difference in kind consists in a difference in the degree of mutual repulsion which two particles exercise, according to the kinds taken. It is further premised that the assemblage is agitated, so as to render unstable all but the final equilibrium arrangement; and a means is provided for linking the particles symmetrically, and unlinking them, under certain circumstances, so as to modify the repulsion between the particles affected. The data thus summarised may be regarded as merely provisional, because the making of the equilibrium arrangement one in which "closest packing" prevails, is the object primarily aimed at; and these concepts are mere devices for attaining this end. By the employment of particles of different kinds, a large amount of variety is provided for. The first step taken is to deduce the law of "closest packing," which runs thus:—Every assemblage of mutually-repellent particles will continually approximate to, or strive after, that relative arrangement of the particles composing it, in which it has come, at every part, to occupy a minimum of space under a given general pressure, or average repulsion, between the particles. This law acts on all assemblages of the nature defined, however numerous the kinds of particles composing them; but for its effects to be traceable, a very limited number of kinds must be present. Passing from assemblages consisting of a single kind of particle, the author takes a very simple case of two kinds of particles confined to a plane, and shows what type of symmetry will be produced when equilibrium is realised. Very simple cases of particles in space are then taken, and it is shown that a large number of different kinds of symmetry are displayed by the equilibrium arrangements

produced when there is variety in the relations between the repulsions. To illustrate "close packing," stacks of balls of various sizes are employed; but it is pointed out that the conditions of statical equilibrium of the particles are not always adequately expressed in this way; although every case of the latter kind can be represented approximately by a case of the former kind, possessed of the same order of symmetry. Very slight variation in the relations between the repulsions alters the form of the equilibrium arrangement; sometimes merely changing the angle without affecting the type; sometimes, when it passes some critical point, bringing about an alteration in type. Changes of the first kind resemble the change in crystal form caused by variation of temperature; whilst those of the latter kind, especially when associated with re-arrangement of the particles, are analogous to polymorphism. In many cases the arrangement of the particles is such that some may be removed without affecting the distribution of the remainder, and without destroying the "close packing." If, therefore, other particles, exercising a slightly less repulsion, be substituted for the removed, *inoperative*, particles, the only resulting change consists in a diminution of the pressure on the particles surrounding them. A species of isomorphism is in this way realised.

When the particles of an assemblage are partially connected by hypothetical linking in a symmetrical manner, similar groups are formed; but, in order that the formation of such groups may not be arbitrary, the partitioning which is produced must have as complete symmetry as that of the partitioned structure. In consequence of this, some kinds of groups are not directly obtainable by symmetrical partitioning of a homogeneous structure; but it is always conceivable that they may be included in the larger groups of some more complex constellation, and that they may be subsequently separated to form an assemblage by themselves. Consequently, very intricate results may be reached by successive steps. Symmetrical intermixture, linking, and unlinking, succeeding one another until complicated groups are built up, for the production of which such an agency as "close packing" appears at first sight inadequate. Having called attention to a large number of arrangements, some capable and some incapable of symmetrical partitioning into groups of a single kind, some linked and some unlinked, the author contends to have established the following two propositions:—
(1) The nature of the symmetry displayed by a homogeneous assemblage of mutually-repellent particles of different kinds in equilibrium, depends on the relations subsisting between the repulsions exercised by these particles.
(2) The assemblages belonging to all of the thirty-two classes of crystalline symmetry result from the fundamental law of "close packing," when the relations between the different repulsions take the widest possible range of variety. Links which restrain the action of the repulsion can be present between some of the particles in some cases. The author refers to crystal "twinning," and points out that the action of dimorphism is competent to produce analogous "twinning" of symmetrical assemblages of linked particles. A number of other properties of linked assemblages analogous to those of crystals are also described. In the domain of chemistry the author cites the continually accumulating experimental evidence of the existence of geometrical arrangement in the molecule, both that established stereochemically and that derived from the study of isomerism, as revealing a state of things precisely such as is arrived at by the law of "closest packing" in assemblages afterwards broken up into similar groups of particles. Attention is called to many groupings of the latter order fulfilling very exactly the conditions of disubstitution in the case of many carbon compounds. While he does not regard his work as throwing any light on the nature of change of state, or change of bulk, the author observes that the distribution in precise proportions of the constituents that must ob-

viously accompany or precede a chemical combination, may fairly be claimed as a resemblance to the regular intermixture brought about according to the law of "closest packing." He further suggests that the reason why some bodies do not readily interact may be due to the "close packing" of one or both.

Prof. HERSCHÉL said he was particularly pleased with the models. He thought it probable that a very wide application would be found for the author's results. There was no doubt much to be learnt from models built up of spheres of two or more sizes, but it would be necessary to learn a great deal more about these symmetrical arrangements before they could be applied with any degree of certainty.

Mr. FLETCHER said it was impossible to criticise the paper without long and careful study. From certain hypotheses the author had deduced a law of "closest packing" that seemed adequate to explain many results observed by chemists and crystallographers; at the same time admitting that the law might be presumed from other reasoning. By his models he had tried to present a picture not of the forms of atoms or molecules, but merely analogical representations of the probable structure of particles. Hitherto the research had been confined to determining the possible arrangements of particles all of one kind, but here were examples of packed spheres of various sizes. It was not quite clear how, in an elementary substance, there could be such a structure, although there certainly were cases of polymorphism awaiting explanation, as for instance with sulphur. The paper, with its 188 pages of MS., represented a vast amount of clear thinking, and many years of admirable work.

Prof. ADAMS called the attention of Fellows of the Physical Society to the Museum at King's College, where were the original models as made and used by the early investigators of this branch of Physics.

Prof. MIERS (communicated, too late for reading)—The principle of "close packing" was not new, but Mr. Barlow was the first to extend it to explain solution, diffusion, and stereochemical problems. His remarks on the growth of curved crystals, vicinal faces, and pseudosymmetrical crystals were open to criticism. With regard to vicinal faces, however, lencite seemed to be a mineral in accord with his hypothesis. The author regarded a crystal as consisting of mutually repellent particles of different sorts: this seemed a very right way of attacking the problem of crystal structure, and would explain some recent observations of Rinne on crystals consisting of water particles and silicate particles. Further, Mr. Barlow had considered the way in which an assemblage might be broken up by the loosening of the ties, and the change of partners, among individual members. That is to say, he had considered crystallisation and solution, features quite ignored by ordinary theories. His view of crystal structure failed to explain why crystals should have faces, and gave no hint as to the controlling forces which keep mutually-repellent particles together. Nevertheless it suggested, among other striking analogies, those bearing on the relationship between crystal structure and chemical constitution, and the irregularities of crystals, such as were commonly neglected in accepted theories. Mr. Barlow had opened up a very promising line of inquiry.

Mr. BARLOW, in replying, said he greatly appreciated the interest shown in his work.

The PRESIDENT then proposed a vote of thanks to the author, and the meeting was adjourned until March 26th. At the invitation of Dr. S. P. Thompson, the Society will on that occasion meet at the Technical College, Leonard Street, Finsbury.

EDINBURGH UNIVERSITY CHEMICAL
SOCIETY.

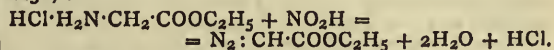
Last Ordinary Meeting of the Session, March 8th, 1897.

Mr. C. SAINTSBURY in the Chair.

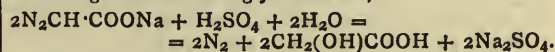
Dr. J. E. MACKENZIE read a paper "On the Compounds of Nitrogen and Hydrogen."

Five such compounds are at present known:— NH_3 , ammonia; N_2H_4 , hydrazine; N_3H , azoimide; N_4H_4 , ammonium nitride; and N_5H_5 , hydrazine nitride.

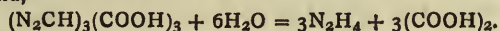
Ammonia does not call for description here. The other substances are of comparatively recent discovery, the chemist to whom we owe most of our knowledge of them being Theodor Curtius. In 1883 he commenced a series of researches on amido-acids. In the course of these he obtained the hydrochloride of ethyl amido-acetate, a beautiful crystalline substance, which, on being diazotised, yielded ethyl diazo-acetate (*Ber.*, xvi., 2230):—



By similar treatment other amido-acid esters yielded diazo esters (*Four. Prak. Chemie*, [2], xxxviii., 404), these being oils, slightly soluble in water, miscible with the ordinary organic solvents, having a characteristic odour and being volatile. The diazo-acetic ester, on treatment with dilute caustic soda, gave the sodium salt, which, on being acidified with dilute sulphuric acid, split off nitrogen and formed glycollic acid,—

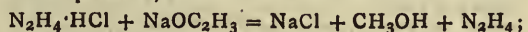


On the other hand, if concentrated caustic soda were used, a polymer, triazo-acetic acid, was formed, which, by the action of acids, broke up into hydrazine and oxalic acid,—



Thus, in 1887, the sulphate of hydrazine was obtained. Other salts were also formed, and from them Curtius separated hydrazine hydrate, $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$, by distilling with strong bases such as CaO or NaHO . The hydrate is a colourless, fuming liquid, b.p. 119° . It destroys cork and rubber, and is a powerful reducing agent and poison.

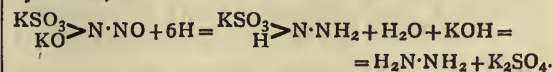
In 1895 Lobry de Bruyn isolated free hydrazine:—
(a) by mixing anhydrous solutions of hydrazine hydrochloride and sodium methylate, separating the sodium chloride and fractionating the resulting solution under reduced pressure,—



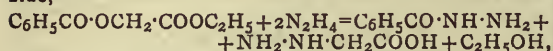
(b) by dehydrating the hydrate by means of barium oxide and distilling under reduced pressure.

Hydrazine melts at $+1.4^\circ$ and boils at 113.5° , under 761 m.m., or at 56° under 71 m.m. pressure of mercury. It is one of the strongest reducing agents. In chlorine it takes fire spontaneously, HCl and N being formed. With sulphur it reacts in cold, with production of SH_2 and N . It displaces ammonia from solutions of ammonium salts. With acids it forms two series of salts, e.g., $\text{N}_2\text{H}_4 \cdot \text{HCl}$ and $\text{N}_2\text{H}_4 \cdot 2\text{HCl}$. Its condensations with aldehyds and ketones are very important.

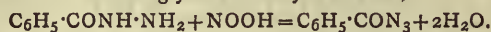
Other methods of preparing hydrazine are those of Pechmann (*Ber.*, xxviii., 1847 and 2374), Thiele and Duden (*Ber.*, xxvii., 3498). That of the latter is the simplest. By the action of sulphurous acid on potassium nitrite, potassium dinitroso-sulphonate is formed, which on reduction by sodium amalgam eventually affords hydrazine,—



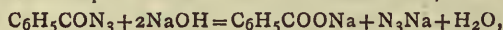
In 1890 Curtius published his first paper on "Azoimide, N_3H " (*Ber.*, xxiii., 3023). By means of hydrazine benzoyl-glycollic ester is converted into benzoyl hydrazide,—



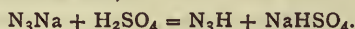
which on diazotising yields benzoyl azoimide,—



This decomposes when boiled with caustic soda,—

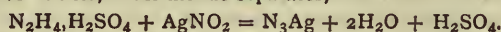


forming sodium nitride, from which azoimide is set free by the action of acids,—



By careful fractionation a solution of 91 per cent N_3H was obtained, which was dehydrated by calcium chloride. Azoimide is a colourless liquid with an unbearable odour, boils at 37° , and is miscible with water and alcohol. It is frightfully explosive; 5 c.grms. exploded in a barometer tube shattering the glass to a powder and spreading the mercury as a fine dust.

Angeli's method of obtaining azoimide is probably the simplest. On mixing solutions of hydrazine sulphate and silver nitrite, silver nitride separates,—



Thiele's method, starting from amido-guanidine, and Wislicenus's from sodamide, are also important.

The metallic salts of azoimide resemble those of hydrochloric acid very much, except that they are explosive. Ammonium nitride, N_3NH_4 , is the most perfect explosive known; 1 kilo. is calculated to liberate 1148 litres of gas at 0° and 760 m.m. (*Bull. Soc. Chim. Paris*, xi., 744).

Hydrazine nitride, $N_3N_2H_5$, is formed by mixing hydrazine hydrate and ammonium nitride or free azoimide. It is a crystalline substance, which behaves like gun-cotton, burning quietly on exposure to flame, but exploding on detonation or sudden heating.

(when genuine) is perhaps the best and purest sugar in the world (p. 148). On the opposite page it is mentioned that sugars having a large bold crystal are not beloved by the trade, as they oppose certain difficulties to the mysterious process known as handling—which, by the way, avenges itself on the men by whom it is effected.

An entire and ably written chapter is devoted to water and its impurities. It is admitted that soluble mineral matter may be tolerated by the brewer to an extent larger than would be tolerated in a drinking or culinary water, or in that employed in the tinctorial art. The injurious action of salts of magnesium in appreciable quantities, we submit, can scarcely be contested. Nitrites in a brewing water he objects to on the authority of Emile Laurent.

The author lays little weight upon Prof. Frankland's previous sewage pollution. He attaches more weight than does Prof. Wanklyn to the possible presence of phosphoric acid in waters, and he considers that waters which after boiling and filtration—we would add *by the Chamberland-Pasteur process*—develop organisms with the Heisch test, probably contain organic matter of an albuminous character, and should be used (if at all) with great caution.

The author quotes the conclusion of Jørgensen, that for brewing purposes it is only necessary to know whether the water contains organisms capable of developing in wort or beer.

Mr. Wright reminds his readers that the presence of free carbonic acid in waters is by no means a proof of purity.

In speaking of materials used as partial substitute for malt, the author mentions that beet-sugar is far inferior to cane-sugar for brewing purposes.

This book will be found most useful not merely to brewers, whether learners or practitioners, but to technologists in general.

NOTICES OF BOOKS.

A Handbook for Brewers; being a Practical Guide to the Art of Brewing and Malting, embracing the Conclusions of Modern Research which bear upon the Practice of Brewing. By HERBERT EDWARDS WRIGHT, M.A., Author of a "Handbook for Young Brewers." Second Edition, thoroughly Revised. London: Crosby Lockwood and Son, 7, Stationers' Hall Court, Ludgate Hill. 1897. Small 8vo., pp. 516.

THIS work is of an extremely comprehensive character. It is concerned not merely with the chemistry of the brewing process, but also with the mechanical construction of brewery plant in the widest sense of the term. Micro-biology and the use of the microscope are very carefully considered. We cannot, however, agree with the author's rather unfavourable estimate of immersion lenses. Several little precautionary hints are given which prove the author to be a practical microscopist.

The controversies between Liebig, Pasteur, Traube, and Brefeld are fairly expounded.

The commercial phase of the brewer's business is next expounded.

As regards the theoretical considerations, Mr. Wright ventures on the opinion that "the final limit of subdivision has probably never been reached." The "pleomorphic craze" of A. G. Salamon and others meets with little countenance. But such questions have a very subordinate interest for the brewer, or indeed for any technical chemist.

We are glad to find the admission that Demerara sugar

CORRESPONDENCE.

LOST PLATINUM.

To the Editor of the Chemical News.

SIR,—Three platinum basins have recently disappeared from this laboratory, and, as it is just possible that some amongst your numerous readers may have been buying second-hand platinum basins, I append the following particulars, in the hope—perhaps somewhat remote—that the platinum may be recovered and the thief brought to book: Basin marked A (scratched on the side), weight 21·877 grms.; basin scratched B, weight 22·412 grms.; basin scratched X, weight 59·747 grms.—I am, &c.,

S. ARCHD. VASEY.

Laboratory, 423, Strand, W.C.,
March 17, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 8, February 22, 1897.

M. Violle has been elected a Member of the Academy in the Section of Physics, *vice* M. Fizeau, deceased.

M. de Heen, of Liège, addresses to the Academy two notes, entitled "Existence of Anodic Rays analogous to

the Cathodic Rays," and "Photography of the Electric Radiations of the Sun and of the Solar Atmosphere."

M. Breton demanded the opening of two sealed papers recently deposited by him, and relating, the one to "the use of alternating simple currents, diphasic and triphasic, for the production of X-rays;" and the other to "a radiographic phial for the refrigeration of the anticathode by a current of cold water."

M. de Sanderval addressed a paper, accompanying photographs obtained through metallic plates of different natures.

M. A. Graby, of Malange, sent the description of a new photographic procedure, rendering it possible to obtain positives in the colours without the production of a proof.

New Method for producing Transparent Crystals.—Ch. de Wateville.—If during its growth we give to a crystal a movement of rotation on itself it assumes a transparency and a lustre analogous to those of precious stones seen cut and polished whatever may be the axis of the crystal near which the rotation is effected. The movement appears not to lose any influence upon the relative development of the faces unless it is very rapid and the solution is very concentrated. If we operate, *e.g.*, upon a solution of alum saturated above 50° (and with the speed of several rotations per second), we progressively disappear the faces of the dodecahedron, and those of the cube which the crystal presents at the outset of the operation; those of the octahedron of the maximum density alone remaining. The author has obtained especially satisfactory results with potassium and ammonium alums, copper sulphate, and sodium chlorate.

On Persulphuryl Chloride.—A. Besson.—This memoir will be inserted *in extenso*.

Anethol and its Homologues.—Ch. Mouet and A. Chauvet.—The author describes a new synthesis of anethol, simpler they consider than the procedure of Perkin. They have thus obtained two homologues of anethol (para-butenyl anisol and para-isopentenyl anisol).

Soluble Oxidising Ferment of the Cassage of Wines.—P. Cazeneuve.—Not suitable for useful abstraction.

Detection of Coal-tar Colours in White Wines and the Difference between the Colours and those of Caramel.—Alb. d'Aguiar and W. da Silva.—This memoir will be inserted in full.

Revue Universelle des Mines et de la Metallurgie.
Series 3, Vol. xxxvii., No. 1.

This issue contains no chemical matter.

No. 2.

Ores of Manganese in Russia (*Chemiker Zeitung*).—It appears that in 1896 the Russian output of manganese ores was 248,000 tons, that of the rest of the world being only 66,821 tons.

Bulletin de la Société d'Encouragement pour l'Industrie Nationale. Series 5, Vol. ii., No. 1.

Review of the Progress effected in the Industry of Essences and Perfumes.—A. Haller.—The author claims the production of perfumes as an art evidently French. Only the English products approach them in fineness, but they possess neither the distinction nor the delicacy of French perfumery. France, both on its southern coasts and in Algeria, possesses great natural advantages for this manufacture; but he recommends that the aid of chemical science should be carefully and eagerly sought for. He raises the question whether the French madder production might not have been saved by such means, pointing out that, in spite of artificial indigo, the yield of India has since 1886 been increased

from 33,320 chests yearly to 40,510. The firm Schimmel and Co., of Leipzig, employs at present nine chemists. The English lavender (of Mitcham) cannot be estimated solely according to its percentage of linalyle acetate, and it is yet preferred to common essences of lavender which contain from 30 to 40 per cent. We cannot help expressing our deep regret that a part of the precious Mitcham soil has been allowed to fall into the hands of "Jerry." Whether the oil of lavender obtained at Sandy (Bedfordshire) is equal to the Mitcham growth we are not yet able to decide. English essence of peppermint (Mitcham) is always in demand on account of the fineness of its odour, the cause of which chemical research has not yet been able to detect.

MISCELLANEOUS.

The Chemiker Zeitung.—The issue of February 20th gives a plan and a full description of the Pharmaceutical Institute and Laboratory for Applied Chemistry in connection with the University of Munich. This journal is also discussing the possible influence of the deposit of "sealed papers" describing the details of an invention may have upon a patent subsequently obtained.

Action of Nitrogen Oxides upon Ferrous Chloride and Bromide.—V. Thomas.—The compounds obtained are inalterable in dry air, and undergo no loss of weight in a vacuum. In most cases they are capable of being split up into chloride (or bromide) and nitrogen peroxide. —*Comptes Rendus*, cxxiv., No. 8.

Action of Dilute Nitric Acid upon Nitrates in Presence of Ether.—M. Tanret.—On agitating water containing nitric acid with aqueous ether, the acid is distributed by the water and the ether so that the quantities dissolved by an equal volume of each liquid bear a constant relation to each other. This relation has been called by MM. Berthelot and Jungfleisch the *coefficient* of distribution; it is independent of the relative volume of the two liquids, but varies with the temperature and concentration. —*Comptes Rendus*, cxxiv., No. 9.

Discharge of the Röntgen Rays: Part Played by the Surfaces Struck.—Jean Perrin.—The gas effect is readily explained if we admit that the X rays at every point of their track liberate equal quantities of positive and negative electricity, movable along tubes of force which contain them. Similarly the metallic effect is readily explained by supposing that on the contact of a conductor, and in a manner variable with nature, the ionisation of the gas is very intense. I propose to call this phenomenon the superficial ionisation of the gas on contact with the conductor. —*Comptes Rendus*, cxxiv., No. 9.

The Chemical Laboratory of Wiesbaden.—The Chemical Laboratory of Prof. Dr. R. Fresenius has been attended by fifty-eight Students during the Winter Session of 1896-97. Of these, forty-three were from Germany, four from England, two from Switzerland, two from Sweden and Norway, two from the United States of North America, one from Austria, one from Roumania, one from Russia, one from Spain, and one from Brazil. There were three Assistant Demonstrators in the several teaching departments, and twenty Assistants in the Versuchsstationen (private laboratories). Besides the Director, Geh. Hofrath Prof. Dr. R. Fresenius, there are engaged, as Teachers in the Establishment, Prof. Dr. H. Fresenius, Dr. W. Fresenius, Dr. E. Hintz, Dr. med. G. Frank, Dr. W. Lenz, Dr. L. Grünhut, and Architect Brahm. The next Summer Term begins on the 26th of April. Throughout the Winter Session, besides various scientific researches, a great number of analyses have been undertaken in the different departments of the Laboratory for manufacturers of all kinds, in judicial cases, and in many branches of trade, mining, agriculture, and hygiene.

NOTES AND QUERIES.

* * Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Dental Alloys.—(Reply to "Jack").—Consult "Hunter's Mechanical Dentistry" (Longmans and Co., 3s. 6d.). Supplied by Elliot Stock, 62, Paternoster Row, E.C., for 3s. 1d.—G. A. KEYWORTH, Hastings.

MEETINGS FOR THE WEEK.

MONDAY, 22nd.—Society of Arts, 4.30. (Cantor Lectures). "Alloys," by Prof. W. Chandler Roberts-Austen, F.R.S.
TUESDAY, 23rd.—Royal Institution, 3. "Animal Electricity," by Prof. A. D. Waller, F.R.S.
WEDNESDAY, 24th.—Society of Arts, 8. "The Transmission of Power by Alternating Electric Currents," by W. B. Esson, M.Inst.C.E.
THURSDAY, 25th.—Chemical & Pasteur Memorial Lecture, by Prof. P. F. Frankland, F.R.S.
Royal Institution, 3. "The Relation of Geology to History," by Prof. W. Boyd Dawkins, M.A., F.R.S., F.G.S.
Society of Arts, 8. "The Cultivation and Manufacture of Rhea Fibre," by Thomas Barraclough. (This meeting will be held at the Imperial Institute).
FRIDAY, 26th.—Royal Institution, 9. "Early Man in Scotland," by Sir William Turner, F.R.S.
SATURDAY, 27th.—Royal Institution, 3. "Electricity and Electrical Vibrations," by Right Hon. Lord Rayleigh, M.A., F.R.S.

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1948.

ESTIMATION OF THORIA.

CHEMICAL ANALYSIS OF MONAZITE SAND.*

By CHARLES GLASER.

SINCE the introduction of the Auer-Welsbach light, the commercial importance of monazite sand has grown greatly, and chemists are now asked to determine the percentage of true monazite, and especially that of thoria, in samples of the sand. This has heretofore been accomplished indirectly; the quantities of iron, titanium, and silica were determined, and the remainder of the material calculated as monazite. A sample treated in this manner gave the following results:—

Iron oxide	3.50	per cent
Titanic acid.. .. .	4.67	"
Silica	6.40	"
Monazite, by difference	85.43	"
100.00		

The sample contained 18.38 per cent phosphoric acid, which calculated as cerium phosphate (factor 3.32) equals 61.10 per cent.

From analyses printed in Dana's "Mineralogy," it was inferred that after elimination of rutile and silica, the remainder would be found to consist chiefly of phosphates of the cerium group, but this is not true.

For the determination of the actual composition of the monazite sand in question, it was decided to attempt an estimation of each of its components, by means of methods to be found in the available literature. As chief sources of information, Graham-Otto's "Chemistry" and Crookes's "Select Methods in Chemical Analysis" were used; due regard was also given to the work which has appeared in the chemical journals of recent years. I was not able, however, to make an exhaustive examination of the literature.

It became evident that no reliable method could be worked out until examination had been made of all the work which had been done in the field, and it seemed necessary to investigate the whole question. In the following statements of preliminary experiments a large portion of analytical data has been omitted, because otherwise this paper would have been bulky. Only the outlines of a general plan of procedure will therefore be given.

So far as possible, it was my intention to examine all the methods proposed for estimation of thoria, but in one notable instance this could not be done. In the *American Chemical Journal* (vol. xvi.) L. M. Dennis and F. L. Kortright describe a method for estimation of thoria by means of potassium hydronitride, KN_3 . An attempt to work by the method proved a failure in my hands, partly because of a mishap while preparing the reagent, only enough of which was saved for a single qualitative reaction; but chiefly because Mr. Dennis declined, when requested, to give me further information. He replied that he was not then at liberty to detail his experience, "as the potassium hydronitride process is more than an analytical one. It is a commercial process for the preparation of pure thoria, which is, I think, unequalled by any of the methods employed by the Welsbach chemists, Shapleigh included. Some of them have tried to use the method and have failed. I think I know why they failed.

But I do not think it quite fair for them to ask me to help them out of their difficulties.

Although the publication was made in a scientific journal, it seems to have been but a partial statement. For which reason criticism is invited and the value of the work is thrown somewhat in doubt. No further attempt was made to follow it out.

By means of fusion with alkali carbonates, an attempt was made to separate monazite sand into two parts. According to Wöhler, all titanic acid ought to become soluble provided the fusion is made at a sufficiently high temperature. Therefore a blowpipe was used. In later work I employed the highest temperatures afforded by a muffle, and for as many as two hours. But at no time was more than a fraction of the titanic acid rendered soluble in water. Moreover, Wöhler's directions to pour the fusion upon an iron plate, and afterwards to powder it, are not practicable because of loss likely to ensue. It was found best to let the fusion soak in water over night, sometimes even for several days, or until perfect disintegration resulted. But such a procedure may have decreased the solubility of titanic acid in water. Phosphoric acid and alumina (and also silica to a large extent) were completely dissolved out of the fused mass. The portion insoluble in water was rendered soluble by the well-known treatment with strong sulphuric acid, and also by fusion with acid potassium sulphate. The solution thus obtained, after being freed from silica, was boiled to separate titanic acid, from four to seven hours during the first experiment. Later, after addition of sodium sulphite, this was accompanied by saturating with hydrogen sulphide, first in the hot and then in the cooled solution. This method is preferable to the first.

After separation of titanic acid and the metals of the fifth group, various methods were tried for separation of thoria from the other earths. It was found that the solution must not be strongly acid when treated with ammonium oxalate for precipitation of thoria and the metals of the cerium group, or traces of thoria will remain in solution. It is best to nearly neutralise with ammonia, and to precipitate in boiling solution.

During the earlier experiments some difficulty was found in keeping in solution all of the zirconia, which is accomplished only by a large excess of the reagent, while yttria and glucina readily form soluble double salts. Under these conditions oxalates of the cerium metals precipitate immediately, while thorium oxalate separates upon cooling. Attempts to separate thorium oxalate from oxalates of the metals of the cerium group by filtration of the hot solution gave unsatisfactory results. The oxalates will pass through the filter for a long time. Bumping of the liquid made it impracticable to keep it boiling until the entire precipitate became crystalline. But if large quantities of thoria are to be separated from small ones of the other oxalates the method works well.

After the insoluble oxalates were separated by filtration and were washed with water, they were converted into oxides by heating, and were re-dissolved as sulphates. In this strongly concentrated solution, made nearly neutral by ammonia, an attempt was made to separate thoria from the other metals by boiling with sodium hyposulphite. In no instance was a complete separation effected, but such portions as were obtained proved to be quite pure. The single exception was that in which the whole of the cerium was precipitated, for reasons not ascertained. Attempts were made to free thoria from most of the cerium by fractional precipitation with weak ammonia, but no considerable advantage was gained thereby, since repeatedly the second fraction showed traces of thorium.

To determine the solubility or insolubility of the different substances left in the insoluble residue from fusions, such residue was treated with dilute hydrochloric acid, both cold and hot. The solution was found to contain all the iron and titanium, the larger part of the silica, and about one-half of the earths present; these

* From the *Journal of the American Chemical Society*, vol. xviii., No. 9.

Table of Reactions of the Rarer Earths.

	KOH or NaOH.	K ₂ CO ₃ or Na ₂ CO ₃ .	K ₂ SO ₄ or Na ₂ SO ₄ .	Na ₂ S ₂ O ₃ .	NH ₄ OH.	(NH ₄) ₂ CO ₃ .	NH ₄ Cl.	(NH ₄) ₂ C ₂ O ₄ .	NH ₄ C ₂ H ₃ O ₂ .
Al ₂ O ₃	Precipitate soluble in excess.	Precipitate.	Alums.	Precipitate in boiling almost neutral solution.	Precipitate.	Precipitate.	Precipitate from solution in NaOH.	No precipitate.	Nearly neutral solution boiled gives precipitate of basic salt.
BeO ₂	Precipitate soluble in excess. Re-precipitated when boiled or diluted.	Precipitate with difficulty soluble in large excess and in CO ₂ .	No precipitate, no double salt formed. Soluble with difficulty.	No precipitate.	Precipitate insoluble in excess.	Precipitate easily soluble in excess. Precipitate on longed boiling. Uncertain separation from Al ₂ O ₃ .	Hydroxide dissolves on prolonged boiling. NH ₃ escapes.	No precipitate, double salt easily soluble.	No precipitate, double salt easily soluble.
ZrO ₂	Precipitate soluble in excess.	Precipitate slightly soluble in excess. Precipitate by NH ₄ OH.	Double salt soluble in hot solution. Precipitate of basic salt on cooling, insoluble in HCl.	Precipitate hy-droxide mixed with S.	Precipitate insoluble in excess.	Precipitate soluble in large excess. Re-precipitated on boiling.	No precipitate.	Precipitate soluble in excess.	?
Gado-linite earths.	Precipitate soluble in excess.	Precipitate soluble in excess. After some hours insoluble double salt separates (Yt).	Concentrated solution precipitate soluble in H ₂ O or K ₂ SO ₄ (not Yt). Separated from thoria and cerites.	?	Precipitate insoluble in excess.	Precipitate easily soluble in excess. Precipitate insoluble double salt after some hours (Yt).	?	Precipitate red granular powder (Er) from boiling acid solution.	?
ThO ₂	Precipitate soluble in excess.	Precipitate slightly soluble in excess. Turbid on heating; clears on cooling.	Double salt insoluble in saturated solution K ₂ SO ₄ . Sodium salt, much more soluble.	Only partial precipitation, even on long boiling concentrated solution.	Precipitate insoluble in excess. ThO ₂ precipitates before cerites. Crookes.	Like NH ₄ OH.	No precipitate.	Precipitate nearly insoluble in acids. Soluble in excess, hot; precipitate on cooling. HCl precipitates.	No precipitate prevents completely precipitation by (NH ₄) ₂ C ₂ O ₄ . Partial precipitation by HCl, complete by excess NH ₄ OH.
CeO	Precipitate soluble in excess.	Precipitate slightly soluble in excess.	Double salt insoluble in K ₂ SO ₄ solution.	No precipitate (?).	Precipitate insoluble in excess.	Like NH ₄ OH.	No precipitate.	Precipitate even in rather acid solution. Slightly soluble in large excess of acid. Like CeO.	Oxalate insoluble.
Ce ₂ O ₃	Excess gives flesh - coloured precipitate.	Precipitate nearly insoluble in excess.		No precipitate.	Like KOH.	Precipitate soluble in large excess. Re-precipitated on boiling. White precipitate quite insoluble in excess.	No precipitate.	Oxalate insoluble.	
La ₂ O ₃	Precipitate soluble in excess.	Precipitate nearly insoluble in excess.	Double salt soluble with difficulty.	No precipitate.	Precipitate.	White precipitate quite insoluble in excess.	No precipitate.	Precipitate in neutral or ammoniacal (?) solution.	Oxalate insoluble.
Di ₂ O ₃	Precipitate soluble in excess.	Precipitate nearly insoluble in excess.	Double salt soluble with difficulty.	No precipitate.	Precipitate.	Rose-coloured precipitate quite insoluble.	No precipitate.	Precipitate nearly insoluble in oxalic or mineral acids.	Oxalate insoluble.

consisted of relatively large portions of zirconia and glucina. Thoria seems not to enter into solution, but is left with the remainder of the earths.

An attempt was made to separate thorium oxalate from the mixed precipitated oxalates, by boiling with ammonium oxalate. Such boiling, filtering, and crystallising yielded oxalates, which, after ignition, corresponded to 2.29 per cent of oxides. The earths were, however, of a deep orange colour, and contained both cerium and zirconia. The latter was present because an insufficient quantity of ammonium oxalate had been used in the first precipitation. In the oxalates of the cerium metals found insoluble in the above treatment, the presence of thoria could be distinctly proven by means of sodium hyposulphite, for which reason the work proved unsatisfactory.

To facilitate a comparison of the more important reactions of the elements herein studied, the accompanying table has been prepared partly from their known behaviour, and partly from the results obtained during this investigation.

With the view of obtaining further knowledge of the behaviour of thoria, fragments of Welsbach mantles were subjected to analysis. They weighed 0.6591 grm., which after ignition fell to 0.6552 grm. Prolonged treatment with boiling sulphuric acid left a residue of 0.0883 grm., which became soluble in water after fusion with acid potassium sulphate. The solutions thus obtained were examined by the same method, but separately, as follows:—After neutralising with ammonia the greater part of the free acid, the solutions were heated to boiling and hot solution of ammonium oxalate was added.

In solution I. a precipitate appeared, but dissolved rapidly upon addition of more of the reagent.

In solution II. a slight turbidity appeared, there was no precipitate, and it soon became perfectly clear.

Upon cooling, solution I. yielded a moderate quantity of a crystalline deposit, while solution II. gave a copious one. Both precipitates were collected on one filter, washed, ignited, and weighed. They yielded 0.1124 grm. of thoria.

The filtrate from I. gave a copious precipitate with ammonia, while that from II. gave only a slight one; both of these were washed on one filter, re-dissolved in dilute hydrochloric acid, and again precipitated by ammonia. An excess of ammonium carbonate entirely dissolved the precipitate. Potassium hydroxide gave a precipitate not soluble in an excess of the precipitant, indicating zirconia, the weight of which was 0.5580 grm. An attempt to purify it from occluded alkali, by again precipitating with ammonia, failed through an accident, in which part of the material was lost. Calculating by difference, the weight of zirconia ought to have been 0.5428 grm. Both precipitates were pure white.

Therefore, this analysis afforded the following composition of the mantles:—Thoria, 17.15 per cent; zirconia, 82.85 per cent.

The separation of the two earths was effected without difficulty, and the thoria was used in the following experiments:—

0.0487 grm. was weighed, dissolved, and mixed with the solution of cerium metals from a previous experiment. The solution was nearly neutralised with ammonia, heated to boiling, a hot solution of ammonium oxalate added, and the mixture allowed to cool. The precipitate was caught on a filter and washed with cold water, extracted in boiling ammonium oxalate solution, caught on a filter, and washed hot; the filtrate was allowed to cool (precipitate 1). The residue was macerated in a hot solution of ammonium acetate, filtered (residue A), and filtrate examined for thoria, as follows:—Hydrochloric acid was added to separate thoria as oxalate, which fell in part only, and the remainder was obtained by sodium hydroxide (precipitate 2). Both these precipitates afforded but a part of the thoria originally weighed, the greater part being held yet with the cerium metals. The method had failed.

The residue (A) upon the filter was reduced to oxide and dissolved as sulphate. After neutralising with ammonia, the liquid was heated to boiling, and there was added an excess of ammonium oxalate with some ammonium acetate; after filtering, the filtrate was treated with sodium hydroxide (precipitate 3).

The precipitates, thus obtained in three fractions, were ignited and found to weigh 0.0774 grm., showing that the thoria was very impure. The greyish mass was fused with acid potassium sulphate, and unfortunately a small fraction of the fused mass was lost. However, from the saved portion a pure thoria, weighing 0.0402 grm., was obtained.

In the next experiment, 0.0343 grm. of thoria and 0.1004 grm. of impure cerium oxide were dissolved as sulphates, and treated with ammonium oxalate and acetate, as for precipitate 3, next above. By precipitating the filtrate with ammonia there was obtained 0.0360 grm. of impure thoria, which after purification weighed 0.0344 grm. Cerium oxide recovered weighed 0.0935 grm.

I desire to call attention to what has been observed frequently during these experiments. If thorium oxalate, held in solution by ammonium acetate, be precipitated by ammonia, the earth so obtained, when washed with the greatest care and re-dissolved in a mineral acid, cannot from an almost neutral solution be again completely precipitated by ammonium oxalate; even if the earth had been ignited after re-solution. It will also be found that a considerable increase has occurred in its solubility in liquids containing much potassium or ammonium sulphate. When enough thoria has been collected, it is my intention to further examine this peculiar behaviour.

(To be continued).

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING FEBRUARY 28TH, 1897.

By WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolis Water Act, 1871.

London, March 10th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 168 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Feb. 1st to Feb. 28th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in a previous report.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 168 samples examined one was recorded as "clear but dull," the remainder being clear, bright, and well filtered.

The rainfall at Oxford during February was 2.41 inches. The average for thirty years being 1.76 inches, we have had an excess of 0.65 inch; 1.48 inches fell on the first five days of the month.

Our bacteriological examination of the London waters give the following results —

	Microbes per c.c.
Thames water, unfiltered (average of 20 samples)	9580
Thames water, from the clear water wells of five Thames-derived supplies (average of 99 samples)	22
Ditto ditto highest	89
Ditto ditto lowest	3
New River, unfiltered (average of 19 samples)	1589
New River, from the Company's clear water well (average of 20 samples)	20
River Lea, unfiltered (average of 19 samples)	1177
River Lea, from the East London Water Company's clear water well (average of 19 samples)	40

The quality of the London waters during February has been uniformly good, with one exception. Since the beginning of this year we have much enlarged the scope of our enquiries, and have examined bacteriologically the clear water well at the Hampton Works of the Grand Junction Water Company, as well as that at the Kew Works of the same Company. On some occasions during the month the Hampton water has given high results; these were at once communicated to the Engineer of the Company, and the matter is receiving serious attention.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.

JAMES DEWAR.

A REVISION OF THE ATOMIC WEIGHT OF MAGNESIUM.*

By THEODORE WILLIAM RICHARDS

and
HARRY GEORGE PARKER.

ALTHOUGH numerous determinations of the atomic weight of magnesium have been made, the results obtained show such very wide variations among themselves that the value in use at present cannot be accepted with any certainty. It will not be necessary to review in detail all the work published, as most of it was done more than forty years ago, before quantitative methods had attained their present exactness; but the following table of methods used and results obtained will assist in a clear comprehension of the situation.

Previous Work on the Atomic Weight of Magnesium.†

Synthesis of sulphate by the action of sulphuric acid on the oxide.

Berzelius, 1826. "Lehrbuch," 5th edition, iii., 1227.	25'3
Svanberg and Nordenfeldt, 1848. Erdmann's <i>Journ. Prakt. Chem.</i> , 1848, xlv., 473.	24'7
Bahr, 1852. Erdmann's <i>Journ. Prakt. Chem.</i> , 1852, lvi., 310.	24'8
Marignac, 1884. <i>Ann. Chim. Phys.</i> , 1884, (5), i., 289, 321.	24'37

Conversion of sulphate into oxide.

Jacquelin, 1851. <i>Ann. Chim. Phys.</i> , (3), xxxii., 195.	24'5
--	------

Determination of sulphuric acid in sulphate.

Gay-Lussac, 1820. <i>Ann. Chim. Phys.</i> , xiii., 308.	24'6
Scheerer, 1846, <i>Pogg. Ann.</i> , 1846, lxi., 535.	24'5
Scheerer, 1847, Later Correction. <i>Pogg. Ann.</i> , 1847, lxx., 407.	24'5?
Jacquelin, 1851. <i>Ann. Chim. Phys.</i> , 1851, (3), xxxii., 195.	24'2

* Contributions from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, vol. xxxii., No. 2.

† We are indebted to Mr. F. W. Clarke for most of the above references.

Conversion of oxalate into oxide.

Svanberg and Nordenfeldt, 1848. Erdmann's <i>Journ. Prakt. Chem.</i> , 1848, xlv., 473.	24'7
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Determination of chlorine in magnesian chloride.

Dumas, 1859. <i>Ann. Chim. Phys.</i> , 1859, (3), lv., 129, 187.	24'6
--	------

Conversion of carbonate into the oxide.

Marchand and Scheerer, 1850. Erdmann's <i>Journ. Prakt. Chem.</i> , 1850, l., 385.	24'0
Scheerer, 1859, Later Correction. <i>Liebig's Ann.</i> , 1859, cx., 236.	24'0

Conversion of metal into oxide.

Burton and Vorce, 1890. <i>Am. Chem. Journ.</i> , 1890, xii., 219.	24'29
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It will be seen that, with the exception of the results obtained by the precipitation of the sulphuric acid with barium chloride and the precipitation of the chlorine with argentic nitrate, all the methods employed involve the use of magnesian oxide. The fact that all such results are untrustworthy was shown by T. W. Richards and E. F. Rogers (*Proc. Amer. Acad.*, xxviii., 200) in their work upon the occlusion of gases by the oxides of certain metals when obtained by the ignition of various salts. The error from this source is so large that it seems hopeless to apply a correction to previous work upon the atomic weight of magnesium, as the amount of gas occluded depends in a large degree upon the method and thoroughness of ignition.

Concerning the results obtained by the precipitation of the sulphuric acid in magnesian sulphate, it is only necessary to point out the error due to the occlusion of various soluble substances present in the solution from which the precipitation was made. This error was recognised by Scheerer, after publishing his results, and an approximate correction was made; but such a correction does not merit much confidence, as will be seen.

In the work of Dumas it is evident that some magnesian oxychloride was formed, and he does not appear at all confident of the accuracy of his results. From the experience of the writers it does not seem likely that the method which he used would give magnesian chloride free from the oxide.

Preliminary Experiments.

Because considerable experience had been gained in a previous research (*Proc. Amer. Acad. Arts Sci.*, xxii., p. 67) upon the occlusion by baric sulphate of salts present in a solution from which this insoluble salt was precipitated, it was thought that Gay-Lussac's and Scheerer's method of precipitating magnesian sulphate with baric chloride might now be used with advantage, applying subsequently the necessary corrections for occluded substances. It had previously been found that the concentration of the solution and the method of pouring had a great deal to do with the amount of occlusion; and hence it seemed likely that by working in a very dilute solution and pouring the magnesian sulphate into the baric chloride with extreme slowness, the occlusion of baric chloride might be large, but that the precipitate might be free from magnesium. Several experiments were made to ascertain the correctness of this supposition, but in each case it was found that, notwithstanding the precautions adopted, a very notable quantity of magnesium was occluded in the baric sulphate. It had been the custom in working upon this precipitation to fuse the weighed baric sulphate with sodic carbonate, to extract the sodic chloride thus formed, and to determine the chlorine with argentic nitrate and calculate as baric chloride, subtracting this amount from the total weight of baric sulphate found. This method gave very satisfactory results, but of course it could not be applied when the baric sulphate was mixed with magnesian chloride and sulphate as well as baric chloride, for then no one could discover the proportion in which each salt was present with sufficient accuracy for work upon atomic weights.

The possibility of obtaining satisfactory results by the determination of the chlorine in magnesian chloride was now considered. The great disadvantage of this method, as is well known, is the extreme difficulty of obtaining pure anhydrous magnesian chloride. The usual method of igniting the double chloride of ammonium and magnesium was tried a number of times, but it was found that a quantity of the oxychloride was always formed. As indicators do not give a sharp reaction in the presence of magnesian salts, the hydrochloric acid driven off cannot be added afterwards by titrating back to the neutral point with a weak acid solution, and it is therefore necessary to obtain in the first place magnesian chloride containing its full complement of acid.

The method was then modified by conducting the ignition of the double salt in a tightly covered platinum crucible in a stream of hydrochloric acid instead of air. That a considerable quantity of oxychloride was usually formed, even under these conditions, was easily ascertained by dissolving the resulting product in water, when the oxychloride remained as an insoluble residue. In two or three cases, however, the amount of oxychloride formed was comparatively small; hence it was hoped that, if the right conditions could be found, the chloride might be obtained in a pure state. Another series of experiments with a modified apparatus was therefore undertaken.

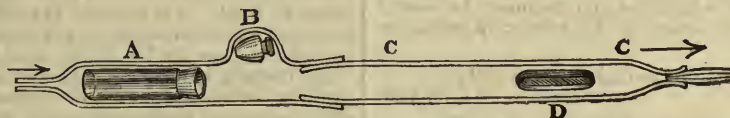


FIG. 1.—BOTTLING APPARATUS, HORIZONTAL SECTION.

A, weighing-bottle. B, stopper of bottle. C C, hard glass tube. D, platinum boat containing fused magnesian chloride.

The expulsion of the ammoniac chloride was conducted in a combustion-tube and the number of drying-tubes was increased, so that the hydrochloric acid gas might be as free as possible from water. The heat was applied very gradually, in order that the double chloride might be almost anhydrous before the sublimation of the ammoniac chloride began. This method gave better results. It was observed that in two or three experiments, where the conditions had been unusually favourable, the resulting chloride gave a clear solution; and it seemed therefore probable that, if an apparatus could be devised to deliver a rapid stream of hydrochloric acid gas entirely free from aqueous vapour, the method might be successful.

Assuming that these conditions might be fulfilled, another difficulty remained to be overcome; for even if the magnesian chloride could be obtained in the combustion tube free from water and oxychloride, the problem still remained to weigh the salt without foreign admixture. If the boat were allowed to remain in the tube until cool, and then removed to a weighing-bottle, the salt must absorb a very notable quantity of moisture from the air in the operation, however quickly this operation might be performed. The boat cannot be transferred to another tube and re-heated, as the moisture present reacts upon the chloride, forming some oxychloride and liberating hydrochloric acid. If it is taken from the combustion-tube while hot and allowed to cool in a weighing bottle, the same effect is produced. Dumas had met with the same difficulties in his work with this method, and he endeavoured to compromise matters by removing the boat from the combustion-tube when it had only partly cooled. As his subsequent results proved, however, the moisture from the air reacted upon the chloride, forming some oxychloride, which interfered seriously with the accuracy of his work. To obviate this difficulty the form of apparatus used by one of us (Richards, *Proc. Amer. Acad.*, xxx., 383) in drying strontic bromide was altered so that the boat could be transferred directly from the ignition-tube to the weighing-bottle without an instant's exposure to the outside air. In order to accomplish this

result the hard glass tube was ground with a long tapering joint directly into the wider desiccating or cooling tube used to contain the weighing bottle. This desiccating-tube had a sort of bulb or "pocket" blown upon one side of it, to receive the stopper of the weighing-bottle, thus allowing the boat to be pushed past the stopper directly from the ignition-tube into the bottle. Afterwards the stopper could be rolled into place with a rod provided for the purpose. The arrangement was used with great success in a recent determination of the atomic weight of zinc (Richards and Rogers, *Proc. Amer. Acad.*, xxxi., 158, 174), to which it was equally applicable. A reference to the annexed sketch (Fig. 1) will make the apparatus more comprehensible.

The desiccating apparatus for the hydrochloric acid gas consisted of two towers, composed of a number of glass bulbs filled with beads, upon which strong sulphuric acid was allowed to trickle from small reservoirs at the top into suitable receptacles at the bottom. This apparatus was constructed wholly of glass, with glass gridirons for flexibility, and ground or sealed glass connections. Joints were made tight with syrupy phosphoric acid (Morley). The hydrochloric acid, after being evolved by allowing strong sulphuric acid to run into a flask containing a strong solution of hydrochloric acid, was passed through a wash-bottle containing sulphuric acid, thence through

the towers just described, afterwards through a tube containing phosphoric pentoxide, and finally into the combustion tube. The apparatus was so arranged that the current of air from an aspirator could be passed through another set of towers, a duplicate of those used for drying the acid gas. By means of stopcocks either dry hydrochloric acid gas or dry air could be passed through the tube containing the weighing tube and boat.

With the help of this contrivance it was found possible to drive off the ammoniac chloride in a current of dry hydrochloric acid, to drive off the excess of acid from the fused magnesian chloride by means of a current of perfectly dry air, and to shut up the pure salt in a weighing-bottle without the least possible means of access of a trace of aqueous vapour. The details of the method will be described later; magnesian chloride prepared after this fashion gives a perfectly clear solution in water. Since this problem was solved, attention was now turned to the preparation of materials for the atomic weight determinations.

(To be continued.)

Industrial Transformation of Oleic Acid into Stearolactone and Monooxystearic Acid.—M. David.—The author can form at will stearolactone and oxystearic acid, or, if the liquid is exposed to cold, all the oxystearic acid may be converted into stearolactone.—*Comptes Rend.*, cxxiv., No. 9.

Procedure for the Determination or Extraction of Gold from Auriferous Ores.—E. Serrent.—The author introduces into the mass of ground ore, in proportions calculated according to the supposed percentage of gold, a mixture of sodium chloride and nitrate with sulphuric acid. When the reaction is completed the gold chloride is dissolved out by the addition of water and the gold is precipitated by ferrous sulphate.—*Comptes Rend.*, cxxiv., No. 9.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

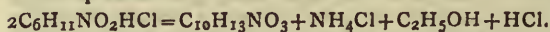
Ordinary Meeting, February 18th, 1897.

MR. A. G. VERNON HARCOURT, President, in the Chair.

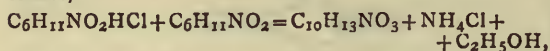
(Concluded from p. 140).

*19. "On the Production of Pyridine Derivatives from Ethylic β -amido-crotonate. By J. NORMAN COLLIE, Ph.D., F.R.S.

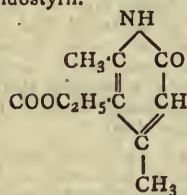
Amongst compounds from which pyridine derivatives can be obtained, ethylic acetoacetate stands out prominently. The author has already called attention to the fact that, when ethylic β -amido-crotonate is distilled, various pyridine compounds are formed. When the hydrochloride of ethylic β -amido crotonate is heated to a temperature of about 120° , it at once condenses according to the equation—



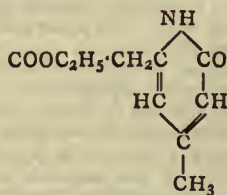
This compound, $C_{10}H_{13}NO_3$, is the ethylic ether of an oxylutidine; it melts at $138-139^{\circ}$. If in its production the hydrochloride of ethylic β -amidocrotonate be heated with one molecular quantity of ethylic β -amidocrotonate,—



an isomeric ether is obtained, m. p. $166-167^{\circ}$. The acids obtained from these two ethers melt respectively at $300-304^{\circ}$ and $190-191^{\circ}$, and both acids decompose at their melting-point, lose carbon dioxide, and give pseudolutidostyryl.



Ether, m. p. 139° .
Acid, m. p. 300° .
A.



Ether, m. p. 167° .
Acid, m. p. 190° .
B.

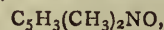
Ether A, when boiled with soda, only hydrolyses with considerable difficulty. It does not react with acetyl chloride, hydroxylamine, or nitrous acids; strong sulphuric acid dissolves it on warming, but the substance is precipitated unchanged when the mixture is poured into water. With bromine, a mono-substituted derivative is produced, $C_{10}H_{12}NO_3Br$, m. p. $158-159^{\circ}$. With phosphorus pentachloride a chloro-lutidine derivative results, $C_{10}H_{12}NO_2Cl$, which is an oil, b. p. $288-290^{\circ}$. After prolonged treatment with tin and hydrochloric acid, the chlorine is removed and replaced by hydrogen, and an α - γ -dimethyl- β -ethylic carboxylate of pyridine, b. p. $246-248^{\circ}$, is obtained.

The acid obtained by the hydrolysis of ether A is very insoluble in water, but can best be re-crystallised from that solvent; various attempts were made to convert this acid into the isomeric acid obtained from ether B, but without result.

Ether B, which is isomeric with ether A, is hydrolysed at once when added to soda solution and warmed. It gives a compound with phenylhydrazine, and when boiled with strong hydrochloric acid is decomposed; it is much less stable than ether A.

With bromine it gives a di-substituted product at once, $C_{10}H_{11}NO_3Br_2$. This compound, when treated with soda, gives the sodium salt of a dibromo-acid, which acid melts at $227-228^{\circ}$ with complete decomposition. Ether B gives

on hydrolysis an acid, m. p. $190-191^{\circ}$, which can easily be crystallised from hot water; when melted it decomposes quantitatively into carbon dioxide and pseudolutidostyryl in exactly the same manner as the isomeric acid, m. p. $300-304^{\circ}$. Pseudolutidostyryl,—



which is a dimethylpyridine derivative, was first obtained by Hantzsch (*Ber.*, 1884, xvii., 2904), by the action of heat on a trimethylpyridine derivative. It was found on heating pseudolutidostyryl with zinc dust, that, although some dimethylpyridine (lutidine) was formed, the chief product of the reaction was a trimethylpyridine (colidine).

Pseudolutidostyryl, when acted on by phosphorus pentachloride, gives α - γ -dimethyl- α -chloropyridine, b. p. $212-214^{\circ}$, and this compound when passed over heated zinc dust yields dimethylpyridine alone.

DISCUSSION.

Dr. FORSTER drew attention to the apparent similarity between the reactions of the pyridine derivatives described by Dr. Collie and certain of the nitrogen derivatives of camphor when Tiemann's formula was employed.

Dr. KIPPING was of opinion that there was no essential difference between Tiemann's for camphor and that proposed by Bredt. He thought that the possibility of the occurrence of tautomerism or stereoisomerism in the compounds described by Dr. Collie should be kept in view.

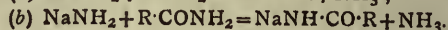
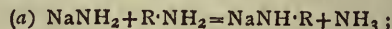
Dr. COLLIE, in reply, said he had not gone completely into the details of the various reactions he had made use of in preparing these substances, and he thought that when the full paper was read it would be seen that the substances were actually different in constitution, and not merely tautomeric or stereoisomeric.

*20. "Sodamide and some of its Substitution Derivatives." By A. W. TITHERLEY, M.Sc., Ph.D.

Sodamide in its reactions with organic haloid compounds invariably gives rise to complex decompositions without appreciable replacement of the halogen by NH_2 . The hydrogen of the sodamide, and not the sodium, reacts, giving hydrochloric acid, which with the amide yields ammonia, whilst the group NaN remains more or less intact, being found afterwards as sodium cyanide and sodium cyanamide. Charring invariably occurs, even when the reaction is conducted with care.

Sodamide on treatment with organic substances possessing a weak acid tendency, such as oximes and hydrazines, readily reacts, giving ammonia and sodium derivatives. In benzene solution these are obtained usually as fine crystalline precipitates, which may sometimes be crystallised from boiling benzene. Sodium acetoxime, sodium hydrazobenzene, sodium phenylhydrazine, and others, have been thus obtained.

A series of substitution derivatives of sodamide formed by the replacement of one or both hydrogen atoms in $NaNH_2$ have also been prepared by the interaction of sodamide with (1) Aromatic amines; (2) Amides, according to the general equations:—



In the former case the reaction is conducted with the substances in the free state in an atmosphere of coal-gas, and in the latter in benzene solution.

Potassium ethylamide, $KNHC_2H_5$, is formed by the careful action of ethylamine gas upon gently heated potassium. On heating, it readily decomposes into potassium cyanide, charcoal, and hydrogen. Sodium phenylamide, $NaNH \cdot C_6H_5$, sodium diphenylamide $NaN(C_6H_5)_2$, sodium p -tolylamide, sodium β -naphthylamide, &c., are all very readily prepared by the above general reaction. They form white, greenish-yellow, or brown, amorphous solids with conchoidal fracture, or light yellowish powders, which are blackened and decom-

posed quickly in the air, darkening especially when moistened with benzene.

When sodamide reacts with organic amides (best in boiling benzene solution) ammonia is rapidly evolved, and the substituted sodamides are obtained as fine, white, crystalline solids, those of larger molecular weight being appreciably soluble in benzene.

Sodium formamide, $\text{NaNH}\cdot\text{CO}\cdot\text{H}$, sodium acetamide, $\text{NaNH}\cdot\text{COCH}_3$, sodium propionamide, and sodium benzamide have been thus prepared—the latter apparently identical with the compound obtained by Curtius from the action of sodium upon benzamide by long-continued boiling in xylol solution.

The latter class of substituted sodamides are soluble without decomposition in alcohol, and their solutions, on treatment with alcoholic silver nitrate, throw down bright orange-red precipitates of the silver compounds, which are very unstable. From the colour of these silver derivatives, and the difficulty with which they and the sodium compounds appear to react with alkyl iodides, &c., the author concludes that the silver and sodium atoms, respectively, are directly attached to nitrogen, and that therefore the above derivatives are to be represented as possessing the ordinary amide and not the imido-hydroxy formula; the amides themselves are most probably tautomeric substances.

*21. "Rubidamide." By A. W. TITHERLEY, M.Sc., Ph.D.

Metallic rubidium behaves like the other alkali metals towards ammonia, displacing one atom of hydrogen and forming rubidamide, RbNH_2 . Though not so energetic as in the case of lithium, the action is very rapid and commences in the cold. On heating in a silver boat to between $200\text{--}300^\circ$, oily drops of the amide quickly form and flow to a liquid in which the metal floats and partly dissolves to a deep blue solution, at once decolourised and converted into rubidamide by the action of ammonia. Rubidamide crystallises in plates melting at $285\text{--}287^\circ$, higher than sodamide and potassamide, but lower than lithamide. At 400° it distils undecomposed in a current of ammonia. With water it is violently decomposed, giving ammonia and rubidium hydrate. Alcohol also decomposes it, and its behaviour with organic substances is very similar to that of sodamide or potassamide.

*22. "On the Spectrographic Analysis of some Commercial Samples of Metals, of Chemical Preparations, and of Minerals from Stassfurt Potash Beds." By W. N. HARTLEY, F.R.S., and HUGH RAMAGE.

In continuation of the work already published (*Roy. Soc. Proc.*, 1896, ix., 393, and *Proc.*, 1897, xiii., 11), samples were examined of steel made at Middlesbrough from the blast-furnace metal smelted from Cleveland clay ironstone, and rolled into rails; of alumina and "red mud" separated from bauxite at the British Aluminium Co.'s Works at Larne, and of the aluminium prepared from the alumina at Foyers and of various commercial alums.

It is shown that of the constituents of the blast-furnace metal, the alkali metals, calcium, copper, silver, gallium, manganese, and lead are present also in the steel, but the chromium and nickel have been removed.

Of the constituents of bauxite, traces of sodium, potassium, calcium, copper, silver, gallium, iron, manganese, and lead are found in the metallic aluminium. These elements are also present in larger quantities in the "red mud," and in addition nickel and chromium are present.

The Alums.—Examined directly, by heating 0.5 grm. of the dried sample in the oxyhydrogen flame, sodium, potassium, rubidium, calcium, and thallium are found as common constituents, and copper, gallium, iron, and nickel as occasional constituents. More interesting results were obtained by examining the precipitates produced by potassium ferrocyanide in solutions, containing 50 grms. of the alum strongly acidified with hydrochloric

acid. These precipitates contained the elements sodium, potassium, rubidium, caesium, copper, silver, calcium, gallium, thallium, nickel, manganese, besides iron, which was also present in the acid radical. The rubidium, caesium, gallium, and thallium lines are strong in some of the spectra, and the results indicate that these elements are almost wholly precipitated by this process. A sample of "aluminoferric" from Messrs. Spence and Sons, Manchester, contained all the elements found in the alums, but in much larger quantities. Of these elements the pyrites furnishes the thallium and also a trace of indium found in a by-product of the manufacture of alum, whilst the other elements were traced to the aluminous minerals, bauxite and shale. The shale was richer in alkalis and gallium than the bauxite, but a sample of French bauxite was richer in silver and lithium than either Irish bauxite or shale.

Samples of Stassfurt minerals were examined in the course of the investigation, and were found to yield spectra containing no lines of rubidium, caesium, gallium, or thallium. These salts gave only weak lines of a few elements besides the lines of the principal elements composing them.

It is pointed out in the paper that the elements found by their spectra actually exist in the specimens, as there is no possibility of them being accidentally introduced, and, furthermore, substances have been examined which have given no trace even of such widely distributed elements as potassium and calcium, and in which the D lines are very weak.

The systematic examination of railway metal by such an analytical method as is here employed might lead to results of practical importance. The method reveals the presence of small quantities of metals such as copper, silver, gallium, and lead, which have not been considered in dealing with commercial irons, and the influences of which upon the physical properties of these have not been studied.

DISCUSSION.

Dr. RIDEAL suggested that the calcium present in aluminium and its compounds might be derived from the vessels employed in the manufacture as well as from the bauxite. He thought it probable that calcium might be present as metal in commercial specimens of aluminium.

23. "Dissociation Pressure of Alkylammonium Hydrosulphides." By JAMES WALKER, D.Sc., Ph.D., and JOHN S. LUMSDEN, B.Sc., Ph.D.

The dissociation pressures of ammonium, ethylammonium, and dimethylammonium hydrosulphides have been determined, as well as the dissociation pressures of mixtures of these substances in pairs. The values obtained for the mixtures fell in every case considerably below the values calculated from the dissociation pressures of the components by the law of mass action. The ratios of the dissociation pressures of these substances, whether simple or mixed, are independent of the temperature, a fact which proves their heats of dissociation to be equal.

24. "Supposed Condensation of Benzil with Ethyl Alcohol. A Correction." By FRANCIS ROBERT JAPP, F.R.S.

The author finds that the compound, described by him in a paper published jointly with Miss Owens (*Trans.*, 1885, xlvii., 90), as formed by the condensation of benzil with ethyl alcohol, is in reality identical with Japp and Miller's *anhydracetonedibenzil*, $\text{C}_{31}\text{H}_{24}\text{O}_4$ (m. p. $194\text{--}195^\circ$), and that its formation was due to the presence of acetone in the "methylated spirit" (alcohol "denatured" with 10 per cent of crude wood-spirit), which was used instead of duty-paid alcohol, in the preparation of the compound. The formula, $\text{C}_{30}\text{H}_{24}\text{O}_4$, ascribed to the condensation compound, requires analytical figures differing only very slightly from those required by anhydracetone-dibenzil.

At the time the paper was published, the authors believed the compound to be identical with Limpricht

and Schwanert's *ethylidibenzoin*, $C_{30}H_{26}O_4$, which Jena stated that he had prepared by the action of alcoholic potash on benzil—the reaction employed by the authors. On the strength of this belief, they proposed to alter Limpricht and Schwanert's formula to $C_{30}H_{24}O_4$, and they further cast doubt on the existence of an acetyl derivative which these investigators had prepared.

The author regrets the publication of these perfectly baseless criticisms on Limpricht and Schwanert's work.

The author is indebted to Prof. Alexander Smith for privately informing him that he had not succeeded in preparing the compound from benzil and alcohol, and thus calling his attention to the matter.

25. "*The Viscosity of Mixtures of Miscible Liquids.*" By T. E. THORPE, F.R.S., and J. W. RODGER.

The authors having measured the viscosity of a large number of liquids, mostly carbon compounds and of very different types, at various temperatures up to the boiling-points under a standard atmosphere (*Phil. Trans.*, 1894, clxxxv.A, 379; 1897, clxxxix.A), have made observations on mixtures of chemically indifferent and miscible liquids, with the view of throwing light on the relation of the viscosity of a mixture to the viscosity of its constituents. A sufficiently comprehensive study of this question would afford answers to many questions of interest. Thus it would settle whether viscosity was related to the number of molecules per unit volume or per unit surface, and would indicate, therefore, how viscosity observations—and indeed all observations which depend upon surface effects—should be treated. It would also indicate whether, in the case of a mixture of a simple and a complex liquid, the values of viscosity gave any indication of the decomposition of molecular aggregates, and how such decomposition was related to dilution and temperature.

On the present occasion the authors communicate the results of a series of measurements made at different temperatures on mixtures of carbon tetrachloride and benzene, methyl iodide and carbon disulphide, and ether and chloroform, the last pair of which they studied on account of the relatively considerable evolution of heat which accompanies their admixture. The methods of observation and of reduction were the same as those previously employed, and the apparatus was identical with that already described (*loc. cit.*).

In no case could the density of the mixture be calculated by the ordinary admixture rule. Carbon tetrachloride and benzene contract on mixing, as already found by F. D. Brown (*Trans.*, 1881, xxxix., 207), whereas methyl iodide and carbon disulphide expand. Ether and chloroform contract considerably.

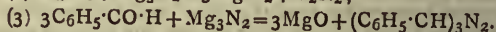
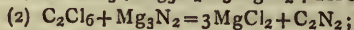
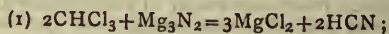
As regards viscosity, the observations afford additional evidence of the fact indicated by Wijkander and supported by Linebarger, that the viscosity of a mixture of miscible and chemically indifferent liquids is rarely, if ever, under all conditions, a linear function of the composition. It seldom happens that a liquid in a mixture preserves the particular viscosity it possesses in the unmixed condition. To judge from the instances hitherto studied, the viscosity of the mixture is, as a rule, uniformly lower than the value calculated on the assumption that each constituent exercises an influence proportional to its amount, although many examples are known to the contrary. No simple relation can as yet be traced between the viscosity of a mixture and that of its constituents.

In the case of a mixture of ether and chloroform, the viscosity at low temperatures is greater than the admixture rule would indicate; but as the temperature is raised, or as the mixture giving the maximum contraction is diluted, the viscosity eventually becomes less than the calculated value, when the general course of the curve showing the relation of viscosity to composition resembles that of such mixtures as carbon tetrachloride and benzene, or of methyl iodide and carbon disulphide. The phenomena in

the case of a mixture of ether and chloroform would seem, to begin with, to be analogous to those of a mixture of ethyl alcohol and water, but the condition which determines the contraction and the maximum viscosity, whether it be a feeble chemical combination or a molecular aggregation of a purely physical character, is destroyed by heat or dilution.

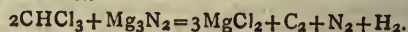
26. "*Magnesium Nitride as a Reagent.*" By H. LLOYD SNAPE, D.Sc., Ph.D.

The object of the experiments detailed in this paper was to investigate whether magnesium nitride could be utilised to introduce nitrogen in the place of oxygen, chlorine, and other negative elements which combine with magnesium. The author investigated the behaviour of magnesium nitride towards chloroform, carbon trichloride, and benzaldehyde respectively, in the hope that the reactions represented by the following equations would occur:—



The substances to be treated with magnesium nitride were sometimes passed in the form of vapour over the latter compound, and sometimes directly mixed with it, the mixture being heated in a sealed tube.

In no case was the desired nitrogenous compound obtained. The chloroform was not attacked at temperatures at which hydrocyanic acid could exist without decomposition, but at higher temperatures an energetic reaction took place, and the observed results were consonant with the reaction—



Carbon trichloride and benzaldehyde were likewise unaffected at temperatures below those at which the anticipated products could be formed. On heating with benzaldehyde to about 240°, a crystalline product, identical with that described by Laurent as amarone, was obtained. Both were subsequently discovered to be identical with the substance named tetraphenylazine by Japp and Burton. (See also the following paper).

27. "*The Identity of Laurent's Amarone with Tetraphenylazine.*" By H. LLOYD SNAPE, D.Sc., Ph.D., and ARTHUR BROOKE, Ph.D.

Amarone being required to compare with the substance obtained, as described in the preceding paper, by the action of magnesium nitride upon benzaldehyde, the authors repeated Laurent's experiments.

It was necessary, in the first instance, to prepare benzoylazotide. This, it was found, could be more readily prepared than by the methods previously given, by the action of ammonium cyanide upon benzaldehyde. Laurent had stated that benzhydramide was produced by the long-continued action of ammonium cyanide upon benzaldehyde, but this was probably due to his having employed an excess of the former reagent. The formula given by Laurent to benzhydramide would accord with its formation by treating benzoylazotide with benzaldehyde, $C_6H_5 \cdot CO \cdot H + C_{15}H_{12}N_2 = C_{22}H_{18}N_2O$. The authors propose to try whether such a reaction can actually be carried out. The vapours of ammonium cyanide were conducted into a mixture of benzaldehyde and alcohol. Crude benzoylazotide slowly separated out, and was washed with alcohol and re-crystallised from benzene. The crystals softened at 198°, and completely melted, with attendant decomposition, at 202°. They were readily soluble in benzene and chloroform; difficultly soluble in alcohol and carbon disulphide, scarcely at all soluble in ether, and insoluble in water. An estimation of nitrogen established their identity with the benzoylazotide previously obtained by other methods.

To prepare amarone, benzoylazotide was next subjected to dry distillation under a pressure of 21 m.m. The residue left, after hydrocyanic acid and other comparatively

volatile vapours had been removed, was crystallised from alcohol containing a small quantity of hydrochloric acid, and washed with some more of the same solution to extract any residual lophine. The crystals which were left melted at 243 to 244°, dissolved in concentrated sulphuric acid giving the characteristic red solution, and behaved towards other solvents precisely in the same manner as the crystalline substance previously prepared from magnesium nitride and benzaldehyde.

By analysis it was found that the empirical formula of amarone was $C_{14}H_{10}N$, not $C_{16}H_{11}N$, as had been stated by Laurent. The amarone which he obtained was evidently not pure, its melting-point being only 233°, or about 10° lower than that of the purified material.

Moreover, a comparison of the properties of pure amarone showed it to be identical with the substance named by Japp and Burton tetraphenylazine, $C_{28}H_{20}N_2$. The authors were kindly supplied by Professor Japp with some of the latter compound, prepared by him from benzoin, for the purpose of instituting this comparison. It was thus established that amarone, as described by Laurent, was actually tetraphenylazine.

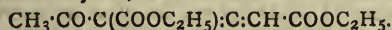
It seems probable to the authors that the substance obtained by Curtius and Blumer having the same empirical formula, $C_{14}H_{10}N$, to which they have not assigned a structural formula, will likewise prove to be tetraphenylazine. The properties of this compound, so far as they have been described, agree with this supposition.

28. "Studies on the Interaction of Highly-purified Gases in presence of Catalytic Agents." Part I. By WM. FRENCH, M.A.

In absence of light, spongy platinum does not appear to bring about combination between oxygen and hydrogen if they have been previously carefully dried; and, so far, experiments seem to show that, after the gases have been in contact with the platinum, added moisture does not cause an explosion.

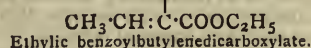
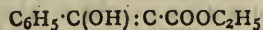
29. "Contributions to the Knowledge of the β -Ketonic Acids." Part III. By S. RUHEMANN, Ph.D., M.A.

The author arrives at the conclusion, from the further study of the action of ethylic chlorofumarate and ethylic α -chlorocrotonate on ethereal salts of β -ketonic acids, that the substances described before (*Trans.*, 1896, lxix., 530, 1383) are to be regarded as ketone-compounds, and he gives the corrections necessitated by the change of view concerning the constitution of the various products there recorded. He further shows that the substance formed from ethylic chlorofumarate and ethylic acetomethylacetate is to be looked upon as ethylic acetoallylenedicarboxylate,—

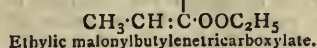
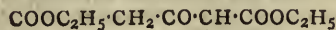


Aniline acts on this ethereal salt with formation of an anile-compound which crystallises in yellow plates (m. p. 180°).

Ethylic benzoylacetate and ethylic acetonedicarboxylate form, with ethylic α -chlorocrotonate, compounds which are to be represented by the formulæ—



and—



The latter substance, under the influence of ammonia, yields two isomeric diamides of the ethereal salt, having the formula $C_{11}H_{16}N_2O_5$, besides a diamide of the corresponding acid.

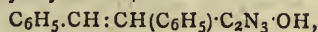
30. "Contributions to the Knowledge of the β -Ketonic Acids." Part IV. By S. RUHEMANN, Ph.D., M.A., and A. S. HEMMY, B.A., M.Sc.

Ethylic acetosuccinate was found to give a colour reaction with ferric chloride, in opposition to the statement of Conrad (*Annalen*, 1877, clxxxviii., 218). The authors give an account of various substances formed from this ethereal salt under the influence of ammonia and of phenylhydrazine. In the latter case, ethylic methylphenylpyrazolone acetate is formed, which, on hydrolysis, yields the corresponding acid. The bromo-derivative of ethylic acetosuccinate was prepared, and on distillation in a vacuum gave ethylic carbottetrinate (*cf.* Moscheles and Cornelius, *Ber.*, 1888, xxi., 2603).

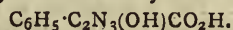
Ethylic benzoylsuccinate, obtained according to Perkin's directions (*Trans.*, 1885, xlvii., 272) was found to distil without decomposition at 192–193° at 10 m.m., and to be decomposed by ammonia with formation of succinamide.

31. "Oxidation of Phenylstyrenyloxytriazole." By GEORGE YOUNG, Ph.D.

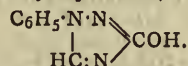
The oxidation by alkaline potassium permanganate of phenylstyrenyloxytriazole,—



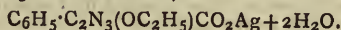
yields phenyloxytriazole carboxylic acid—



This acid, immediately on liberation, loses carbon dioxide and forms phenyloxytriazole,—



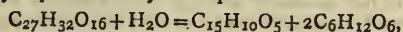
The following derivatives of the carboxylic acid have been prepared:—Ethylic phenylethoxytriazole carboxylate, $Ph \cdot C_2N_3(OEt)CO_2Et$, white needles, m. p. 82–83°. Amide, $C_6H_5 \cdot C_2N_3(OC_2H_5)CONH_2$, white needles m. p. 149–150°. Silver salt,—



Phenylethoxytriazole carboxylic acid, when liberated, loses carbon dioxide and forms phenylethoxytriazole, $C_6H_5 \cdot C_2N_3H \cdot OC_2H_5$, needles, m. p. 60°. This compound has also been formed by the action of ethyl iodide on the silver derivative of phenyloxytriazole.

32. "Apiin and Apigenin." (Preliminary notice). By A. G. PERKIN.

Apiin, a constituent of parsley (*Apium petroselinum*), was first isolated by Braconnot (*Ann.*, 1843, xlviii., 349), and subsequently examined by Planta and Wallace (*Ann.*, 1850, lxxiv., 262), who found it consisted of a glucoside. Gerichten (*Ber.*, 1876, ix., 1124, in a more detailed investigation, assigned it the formula $C_{27}H_{32}O_{16}$, and considered its decomposition by dilute acids to be most probably represented by the equation—



which is based upon the yield of apigenin thus obtained. He described no derivatives of apigenin, but states that by the action of alkali there is produced phloroglucol and an acid, which by prolonged treatment is decomposed with formation of protocatechuic acid, *p*-hydroxybenzoic acid, formic and oxalic acids.

Having suspected, from a description of its properties, that apigenin was a yellow colouring-matter, and this having been proved to be the case, the present investigation was instituted. It is wished to reserve the further study of the reactions of this interesting substance.

The glucoside apiin is somewhat difficult to fully decompose by dilute acids, the apigenin produced after three hours' digestion with hydrochloric acid of sp. gr. 1.04 yielding C=64.3; H=3.90; after ten hours, C=65.81, 65.74; H=3.45, 4.01; and only when so treated for twenty-five hours are numbers obtained indicating the formula $C_{15}H_{10}O_5$, evidently the correct one. Calc., C=66.66; H=3.70; Found, C=66.34, 66.37; H=3.87, 3.81. Apigenin contains no methoxy groups, and does not combine with mineral acids; it, however, forms a

sulphonic acid not yet thoroughly examined. Dibromapigenin crystallises in almost colourless needles, melting above 290° . $C_{15}H_8O_5Br_2$ requires $C=42.05$; $H=1.87$; Found, $C=42.09$; $H=2.23$; and a tribenzoyl compound, $C_{15}H_7O_5(C_6H_5O)_3$, needles, m. p. $210-212^{\circ}$, has also been obtained. Calc., $C=74.23$; $H=3.78$. Found, $C=74.41$; $H=4.17$. Apigenin reacts with diazobenzene, forming $C_{15}H_8O_5(C_6H_5N_2)_2$, orange-red needles, m. p. $290-292^{\circ}$. Calc., $C=67.78$; $H=3.74$; $N=11.71$. Found, $C=67.22$; $H=3.75$; $N=11.54, 11.56$; which yields a monacetyl derivative $C_{15}H_7O_5(C_2H_3O)(C_6H_5N_2)_2$; orange-red leaflets, m. p. $259-260^{\circ}$. Calc., $C=66.92$; $H=3.84$. Found, $C=66.66$; $H=4.05$. Experiments on the further acetylation of this substance are in progress. By treatment with strong alkali there is obtained from apigenin, phloroglucol, an acid, m. p. $208-209^{\circ}$, probably *p*-hydroxybenzoic acid, a trace of acetic acid, and a substance crystallising in colourless needles, m. p. 107° , which bears some resemblance to *p*-hydroxyacetophenone. Fuming nitric acid decomposes apigenin, the principal product being an acid; yellow needles; m. p. $244-245^{\circ}$. The dyeing properties of apigenin will be described in the full communication. The investigation of these substances will be continued, and the study of the ethylation and methylation of apigenin is also in progress.

33. "Note on the Constitution of the so-called 'Nitrogen Iodide.'" By J. W. MALLET, F.R.S.

Mr. Chattaway concludes his paper by saying that "at present the formula NH_3I_2 seems best to accord with the reactions of the compound as a whole, and best to group all the known facts regarding it." Reference to a short paper by my sometime student, Mr. W. H. Seamon, published in the *CHEMICAL NEWS*, 1881, xliv., 188, will show that a very different substance—liquid, and non-explosive—gives results on analysis agreeing well with this formula. It was obtained by the action of dry gaseous ammonia on solid iodine, and appears to be identical with the substance prepared in a different way by Guthrie, of which brief mention is made by Mr. Chattaway in a footnote.

I cannot agree with him that in the explosive compound the ratio of $N:I$ is always $1:2$. Some analytical results of my own (published in the paper on this subject in the *American Chemical Journal*, 1879, i., 4) were quite incompatible with this ratio, and agreed nearly with the ratio $1:3$. In view of the fact that the preparation which gave these results had been freely washed with alcohol and afterwards with ether, I cannot think it probable that any considerable formation and retention of iodoform raised the proportion of iodine.

In any discussion of the composition of the explosive substance in question, some attention ought surely to be given to the probable analogy with nitrogen trichloride, for which Gattermann seems to have fairly well established the formula.

NOTICES OF BOOKS.

Our Secret Friends and Foes. By PERCY FARADAY FRANKLAND, Ph.D., B.Sc., F.R.S., F.C.S., &c., Professor of Chemistry in Mason College, Birmingham. Third Edition, Revised and Enlarged. London and Brighton: Society for Promoting Christian Knowledge. New York: E. and J. B. Young and Co. 1897. Post 8vo. Pp. 238.

THE study and application of bacteriology during the past few years has made such rapid strides, in so many directions, that we are always glad to see new and enlarged editions of such well-known works as those given to us by Prof. Percy Frankland.

This latest addition to bacteriological literature sets orth in a clear, and not too technical manner, the fundamental ideas which govern the study of the science. The

early chapters deal with the micro-organisms in air and in water. Chapters IV. and V. are devoted respectively to useful and malignant micro-organisms, and it is important for it to be as widely known as possible that many microbes are absolutely essential to our existence. There is a widely-spread belief among the general public that *all* microbes are harmful; and great expense, and a good deal of trouble, is incurred in reducing the number of bacteria in water used not only for drinking purposes, but even for watering roads, putting out fires, &c. It should be borne in mind that many of these microbes act in a certain sense as policemen; the microbes of anthrax, typhoid, and such like, cannot live in water "contaminated" with a certain number of harmless microbes. What risk then is run, if perchance a few of these malignant organisms find their way into an almost sterile water-supply where there are not enough "police" microbes to hold them in check!

The new chapter, number VIII., is one of great interest and importance, entitled "Recent Applications of Bacteriology," several recent discoveries of prevention and cure of disease, snake-bite poisoning, &c., being herein described. Is it necessary to add that, owing to the action of our legislators, most of this excellent and valuable work is being done abroad?

Some curious results of experiments on the "manuring" of land by appropriate microbes are described, and an excellent cut is added showing the difference between two similar vetches, *Vicia villosa*, one inoculated with pure cultivations of *Pisum sativum*, nodule-bacteria, and the other uninoculated; the difference is very striking.

At the end of the book is a table giving the number of microbes per c.c. in London waters during the years 1886, 1887, and 1888, wherein we see that, so short a time ago as ten years, the colonies were counted by hundreds, and even thousands, whereas the average number during last year, 1896, was twenty-five.

CORRESPONDENCE.

THE CHEMICAL SOCIETY ELECTION.

To the Editor of the Chemical News.

SIR,—In view of the insidious attempt that is being made to reverse the recommendations of the Council—an attempt absolutely without precedent in the annals of the Society—I would ask you to allow me to bring under the notice of the Fellows generally what I believe to be the true situation.

Although at the outset it was an attack on the distinguished gentleman who has been nominated President by the unanimous vote of a very large and entirely representative Council, I am satisfied that the rancorous opposition of his fellow countrymen is now put quite in the background, and that the majority of those whose names have been given in as nominating another have been led to promise their votes from an entirely different point of view, and not with any personal motives.

Mr. Muir and his immediate following are no doubt determined haters: those who took the trouble to read the letters which disfigured the columns of *Nature* some couple of years ago will be aware of the virulently personal, most ill-advised, and entirely unprofessional attacks which were made on Prof. Dewar, and will be able to rate the opposition of such men at its true value.

Some, no doubt, have been influenced by the suggestion that the work done within recent times by Prof. Ramsay entitles him to the preference, and have joined the movement out of sheer good nature. To these it may be pointed out that, although of a less sensational character, the work done by Prof. Dewar on the physical properties of metals and other substances at very low

temperatures is of altogether extraordinary importance and value in the eyes of those who can appreciate it, and not inferior in interest to any work accomplished during the past few years. Moreover, it has not been initiated by any casual observation, but is the final outcome of preparations made with unwearied perseverance and most remarkable skill during a long series of years, and of work carried out with great courage and at most serious personal risk.

When I ask those about me what motives have inspired their conduct, no two give the same or a straightforward answer. But as I extend my inquiries, I find that wrongheadedness and narrowness, combined with a certain amount of personal vanity, are really at root the cause of the movement.

Dr. Collie, Dr. Kipping and their friends are, in fact, of opinion that undue preference has been shown to senior members of the Society. How the juniors have suffered at the expense of the seniors I am totally unable to discover—the more so when I find that during the past ten years the Society has borne the expense of publishing papers by Dr. Kipping, for example, covering some 500 pages of the Society's *Transactions*. A gentleman who has received such treatment at the hands of the Publishing Committee and the Council can scarcely have any grievous cause of complaint against his seniors—but if he have, let him state it fairly and openly.

I assert, on the basis of considerably over twenty years' experience, that no Council has been more loyal or careful of the interests of all sections of the Society; each year the new members have been selected on the ground of service rendered to Chemical Science and for no other reason, and men such as Collie and Kipping have been given a voice in the management at the earliest possible opportunity.

One of my late students, whose name figures on the list of objectors, to whom I wrote asking his reasons, replies as follows:—

"I allowed my name to be added to the list of Ramsay's nominators on the ground that this is the only means the rank and file of the Society have of influencing the election of Officers, and as a protest against the present mode of election. The present system amounts to the Council electing President and also electing itself.

"I imagine that the fact that Dewar is unpopular has been seized as the means of making the protest."

It is clearly a case in which, as the poet has well put it:—

"The dog to serve some private ends
Went mad and bit the man."

But surely, Sir, Englishmen have a reputation for fairness. It would be outrageous to attempt to damage the public reputation of a man who is deemed good enough by the managers of the Royal Institution to occupy the post of Resident Manager, a post once held by Faraday, and with whom Lord Rayleigh is prepared to work as a colleague in conducting the Davy-Faraday laboratory. Young gentlemen like Dr. Lapworth, but six months ago a student in my laboratory, Mr. Marshall, Mr. Mills, Mr. Evans, all recently students under me, my junior Demonstrator Mr. Pope, Mr. Ling, and many others on the list personally known to me, can neither know anything against Professor Dewar nor have any grievance against the Society which justifies them in their action, and straightlaced representatives of the analytical profession like Mr. Cassal must have other motives than mere personal ones for joining in such a crusade.

Dr. Collie is reported to have stated at the last meeting of the Society that the nomination of Prof. Ramsay had been made without his knowledge or permission. I venture to challenge the accuracy of this statement; if I am not altogether misinformed, Prof. Ramsay has known what is going on from the beginning, and is pledged to "reform" the Society, I believe, by abolishing the "old buffers" and ensconcing the young radicals in their places.

I will yield to no one in the desire in every way to pro-

mote the interests of the Chemical Society as representative of the interests of chemical science in this country. We have accomplished much during the past twenty years, and may do much more in the future; but it behoves us to work together fairly and honestly if we are to continue to exert an influence for good. I trust, therefore, that all right-minded Fellows of the Society will unite in resisting the ill-considered attempt that is being made both to prejudice the position of a man who has the interests of the Society most warmly at heart, and to introduce changes which, whether desirable or not, have not been for one moment previously brought under our notice.—I am, &c.,

HENRY E. ARMSTRONG.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 9, March 9, 1897.

Researches on the Uranic Rays.—Henri Becquerel.—Will be inserted in full.

Discharge of the Röntgen Rays: Part Played by the Surfaces Struck.—Jean Perrin.—Already inserted.

Existence of Anodic Rays Analogous to the Kathodic Rays of Lénard and Crookes.—P. de Haen.—This paper requires the accompanying diagram.

Determination of Atmospheric Ozone on Mont Blanc.—Maurice de Thierry.—Will be inserted in full.

Action of Dilute Nitric Acid upon Nitrates in Presence of Ether.—M. Tanret.—Already inserted.

Action of Aluminium Chloride upon Camphoric Acid.—G. Blanc.—Will be inserted in full.

MEETINGS FOR THE WEEK.

MONDAY, 29th.—Society of Arts, 4.30. (Cantor Lectures). "Alloys," by Prof. W. Chandler Roberts-Austen, F.R.S.

TUESDAY, 30th.—Royal Institution, 3. "Animal Electricity," by Prof. A. D. Waller, F.R.S.

Society of Arts, 8. "Lead-Work," by W. R. Lethaby.

WEDNESDAY, 31st.—Society of Arts, 8. "Cycling—Historical and Practical," by George Lacy Hillier.

Chemical, 3. (Annual General Meeting). Ballot for Election of Officers and Council. At 7 p.m., Anniversary Dinner at the Criterion Restaurant.

THURSDAY, April 1st.—Chemical, 8. "Oxidation of α - γ -dimethyl α -chloro-pyridine," by E. Aston and J. Norman Collie, Ph.D., F.R.S. "Composition of Cooked Fish," by K. I. Williams.

Royal Institution, 3. "The Relation of Geology to History," by Prof. W. Boyd Dawkins, M.A., F.R.S., F.G.S.

Society of Arts, 4.30. "A Visit to Russian Central Asia," by M. F. O'Dwyer.

FRIDAY, 2nd.—Royal Institution, 9. "Metallic Alloys and the Theory of Solution," by C. T. Heycock, F.R.S.

SATURDAY, 3rd.—Royal Institution, 3. "Electricity and Electrical Vibrations," by Right Hon. Lord Rayleigh, M.A., F.R.S.

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1949.

ON THE DETECTION OF THE COLOURING-MATTERS OF COAL IN WHITE WINES, AND ON DISTINGUISHING THESE PIGMENTS FROM THE COLOURS OF CARAMEL.

By ALB. D'AGUIAR and W. DA SILVA.

THE recognition of the coal-tar colours, and their distinction from caramel in liqueurs, has already been undertaken by Rocques, Saglor, Roeser, and other chemists. Quite recently Cruz Nagelhães has sought to demonstrate that the methods in common use for the detection of coal-tar colours in old white wines may prove defective, and lead us to confound caramel with the yellow or orange-yellow colours of coal.

We request permission to present on this question our experiments conducted in the Municipal Laboratory of Oporto.

We shall confine ourselves to reporting the results obtained by the use of amylic alcohol on wines rendered alkaline by the addition of ammonia, with dyeing experiments on silk by immersion in amylic alcohol.

We have experimented with the following colours:—1, binitro-naphthol; 2, chrysoidine; 3, Bismarck brown; 4, orange II.; 5, tropeoline; 6, Biebrich red; 7, azo-flavine; 8, helianthine; 9, methyl-orange; 10, amido-azo-benzene; 11, naphthol yellow S; 12, caramel. These colours were respectively dissolved each in alcohol of sp. gr. 20°, and the solutions were mixed with 400 c.c. of white wine from Ermda, grown in the north of Portugal. The caramel was added to the wine in the proportion of 5 c.c. to 400 c.c., and was obtained with 500 grms. of pure sugar, heated to 215°, and lastly dissolved in 800 c.c. of water. The intensities of colour were compared with Ermda wine, seen in a stratum at the depth of 12 c.m.

We made three series of experiments.

The colours of caramel give, by the usual treatment with amylic alcohol, results very doubtful and sometimes negative. On the contrary, the coal-tar yellows present a group of very distinct colours, which are very characteristic if they are used in the fraudulent wine-trade.—*Chemiker Zeitung*.

ESTIMATION OF THORIA. CHEMICAL ANALYSIS OF MONAZITE SAND.*

By CHARLES GLASER.

(Concluded from p. 147).

Systematic Method of Analysis.

FROM the analytical data given, the following method has been deduced.

It is essential that the mineral be divided to the greatest possible degree. Prolonged powdering in an agate mortar is indispensable. Solution is effected either by prolonged heating with strong sulphuric acid, or by fusion with acid potassium sulphate. In the latter

case, the cooled mass is warmed with so much sulphuric acid that the product, after cooling, may be poured from the crucible. The first method takes more time than the second, but it introduces less of the objectionable potassium salts. It is advisable to fuse only those portions which are insoluble in sulphuric acid.

For estimation of silica the sulphuric acid treatment is preferable, in which case it is best to evaporate once on a sand-bath to dryness, to render silica insoluble, and then to add fresh sulphuric acid. The resulting mixture should be added slowly to ice cold water, which dissolves the mass excepting silica and tantalic acid, with possibly traces of titanio acid, thoria, and zirconia. After filtering, the residue should be ignited and weighed. Silica is eliminated by repeated treatment with hydrofluoric acid. Any residue remaining should be moistened with sulphuric acid, to convert the fluorides of the earths into sulphates, which, after ignition at a high temperature, are weighed as oxides, and silica determined by the loss in weight. The residue of tantalic acid, with possibly traces of the bodies mentioned above, is treated with sulphuric acid and hydrofluoric acid. Tantalic acid remains insoluble, and may be filtered off and weighed. The matter soluble may be added to the main solution.

The original solution is saturated with hydrogen sulphide, first at boiling and then at the ordinary temperature. Titanio acid is precipitated, together with metals of the fifth group. That sodium sulphite assists in the precipitation of titanio acid has not been verified in my work.

When completely settled, the liquid is filtered and the filtrate boiled to expel hydrogen sulphide. Any free acid may be nearly neutralised with ammonia; to the boiling liquid is added an excess of a boiling solution of ammonium oxalate, as much as 100 c.c. of the cold saturated solution for 2 grms. of monazite sand. The excess necessarily must be large. The mixture is then permitted to cool, best for an entire night. The solution will contain phosphoric acid, the oxides of iron, manganese, aluminum, glucinum, yttrium, zirconium, and calcium. In the precipitate will be found thoria and the oxides of the cerium group.

If the bodies in solution are to be estimated, add ammonia to precipitate the metals as phosphates. Filter and wash thoroughly, preserve the filtrate for estimation of phosphoric acid and alumina. Ignite the precipitate and fuse it with mixed carbonates of potassium and sodium. The fused mass is exhausted with hot water, filtered, and the residue well washed with hot water. The filtrate is added to that containing phosphoric acid and alumina.

The remaining oxides and carbonates are dissolved in sulphuric acid and precipitated with ammonia. Lime is estimated in the filtrate therefrom.

When an attempt is now made to dissolve the precipitated hydroxides on the filter by dilute hydrochloric acid, it sometimes occurs that zirconia in part remains. Therefore it is best, after this operation, to incinerate the filter. Then neutralise the solution with ammonia as far as practicable. Pour this slowly, with constant stirring, into a mixture of ammonium carbonate and ammonium sulphide, prepared as follows:—To a solution of ammonium carbonate more than enough to neutralise the free hydrochloric acid above indicated, and to hold in solution the earths to be dealt with, add enough of ammonium sulphide (usually a few c.c.) to precipitate the metals of the fourth group. The latter will be precipitated, while zirconia, yttria, and glucinum remain in solution. Iron and manganese may be determined by the usual methods.

If the carbonate solution be boiled for one hour the earths are completely precipitated. They may be caught on a filter and dissolved in hydrochloric acid; or the carbonate solution may be treated directly with that acid, carbon dioxide expelled by boiling, the solution cooled

* From the *Journal of the American Chemical Society*, vol. xviii, No. 9.

and treated with an excess of sodium hydroxide. Zirconium and yttria are completely precipitated, while glucina remains dissolved; to precipitate this, boil the solution one hour.

To separate zirconia from yttria, dissolve the hydroxides in hydrochloric acid, warm, then saturate the solution with sodium sulphate. When cold, zirconia separates in the well-known manner. From the filtrate ammonia separates yttria.

As the earths are apt to occlude alkali salts, it is best to dissolve and again precipitate them (with ammonia) before they are ignited and weighed.

Separation of the precipitated oxalates of thoria and of the cerium group is accomplished as follows:—The oxalates are reduced to oxides by ignition, then converted into sulphates, the greater part of the free acid neutralised with ammonia, the solution boiled, and boiling ammonium oxalate added in excess. After a short time (as soon as oxalates of the cerium metals have formed, but before the liquid has cooled), a few c.c. of solution of ammonium acetate are added. When cold, the entire cerium group is precipitated as oxalates, while thoria remains in solution. After prolonged standing, best overnight, the insoluble oxalates are removed by filtration; in the filtrate, precipitate thoria with ammonia in excess, filter, ignite, and weigh.

Separation of cerium from lanthanum and didymium is easily accomplished by the well-known method. Pass a current of chlorine through the liquid containing the hydroxides, which have been freshly precipitated by a fixed alkali.

Separation of lanthanum from didymium was not attempted.

An analysis of the monazite sand used in my work, made as indicated in the foregoing notes, gave results as follows:—

Titanic acid	4.67
Silica	6.40
Phosphorus pentoxide	18.38
Lead	trace
Alumina	1.62
Lime	1.20
Cerium oxide (CeO)	32.93
Lanthanum and didymium oxides	7.93
Thoria	1.43
Ferric oxide	7.83
Zirconia and yttria	13.98
Glucina	1.25
Tantalum acid	0.66
Not determined	1.72

100.00

Titanic acid and silica was determined in a separate portion.

The determination of tantalum acid was only approximate, since a part of it is dissolved by fusion with acid potassium sulphate, and thus escapes weighing. As several such fusions were made, it is probable that the greater part of the matter "not determined" ought to be reckoned as tantalum acid. The quantity stated was an average of three determinations (minus or plus 0.05) from the residue of repeated fusions.

Through the courtesy of Mr. H. B. C. Nitze, of the Geological Survey of North Carolina, I have received a number of samples of monazite sand mined at various localities in that state. Two of these had been prepared by a new process, and were found to be practically free from rutile and garnets. They were excellent material for my methods of analysis, and they gave results as follows:—

Analysis of a Coarse Monazite Sand from Shelby, North Carolina.

Silica	3.20
Titanic acid	0.61
Cerium metals as CeO	63.80
Phosphorus pentoxide	28.16
Thoria	2.32
Zirconia, glucina, yttria	1.52
Manganese	trace
No iron, alumina, or lime	0.00

99.61

The colour of this sand was honey-yellow.

Analysis of a Fine Monazite Sand from Bellewood, North Carolina.

Silica	1.45
Titanic acid	1.40
Cerium metals as CeO	59.09
Phosphorus pentoxide	26.05
Thoria	1.19
Zirconia, glucina, yttria	2.68
Tantalum acid	6.39
Iron and manganese oxides	0.65
Alumina	0.15

99.05

The colour of this sand was honey-yellow.

A REVISION OF THE ATOMIC WEIGHT OF MAGNESIUM.*

By THEODORE WILLIAM RICHARDS
and
HARRY GEORGE PARKER.

(Continued from p. 149).

Preparation of Materials.

THE sample of ammoniacal magnesian chloride which will be hereafter referred to as sample No. 1, was prepared as follows:—About 500 grms. of ordinary "C. P." magnesian chloride were saturated with hydrogen sulphide, a small amount of ammonia was added, and the whole was allowed to stand in a warm place for several days. To the supernatant liquid after decantation a small quantity of very pure ammoniacal oxalate was added. The magnesian chloride thus almost wholly freed from calcium was again decanted; and after more ammoniacal oxalate had been added, the whole was allowed to stand, and the clear liquid was yet once more decanted. The solution was then evaporated to dryness, and the resulting cake dried in an oven and ignited in a platinum dish. The mixture of magnesian oxide and oxychloride thus formed was washed with the aid of a filter pump for about sixty hours. At the end of this time, although the wash water contained no sodium, the insoluble precipitate was not free from that metal. The precipitate was therefore dissolved in hydrochloric acid, previously distilled in platinum for the purpose, and the solution was filtered. In order to eliminate the sodium, a portion of the magnesium was precipitated by passing into the solution a current of ammonia gas. The precipitate formed by this very wasteful process was washed for several days, at the end of which time it was found to be free from any appreciable traces of sodium and potassium, when tested with the spectroscope.

Ammoniacal chloride was now prepared by mixing streams of ammonia and hydrochloric acid gas. This gave am-

* Contributions from the Chemical Laboratory of Harvard College, From the *Proceedings of the American Academy of Arts and Sciences*, vol. xxii., No. 2.

monic chloride mixed probably to a certain extent with various amines, but free from inorganic salts. As the amines must be driven off later, it was not thought worth while to take the trouble of removing them at this stage of the work.

The solution of ammoniac chloride thus prepared was added to the solution of magnesian chloride obtained by dissolving the oxychloride in hydrochloric acid in proportions corresponding to formula $Mg.Cl_2(NH_4)Cl$, and the mixture was carefully evaporated to dryness and gently heated in an oven. It is, of course, unnecessary to say that all the latter part of this purification was done as far as possible in platinum. The solid cake was powdered in an agate mortar, and placed in a glass-stoppered bottle which was kept in a closed jar. The double chloride thus prepared was then tested with the spectro-scope, but no impurities could be discovered; and its solution in water was perfectly clear. Tests were made with ammoniac oxalate and baric chloride, but in neither case was a precipitate formed on long standing.

The second sample of magnesian chloride was treated in a similar way up to the point where it was necessary to get rid of sodium and potassium. The solution was evaporated to dryness in a platinum dish with the aid of an alcohol lamp, and the resulting cake was gently ignited and then washed for a long time, nothing but platinum being allowed to come in contact with the material from this time forth, and all the heating being done by means of alcohol lamps to avoid the danger of contamination of sulphur from illuminating gas. The oxychloride thus formed was then dissolved in pure hydrochloric acid and filtered. By evaporating down again the magnesium was again rendered insoluble. This process was repeated again and again, until there was no trace of sodium or potassium remaining.

The ammoniac chloride necessary for the preparation of the double salt from this second sample of magnesian chloride was prepared by digesting ammoniac chloride with nitric acid to destroy the amines (Krüss, *Liebig's Annal.*, cccxxviii., 51). It was then dried, sublimed several times, re-crystallised five or six times from its aqueous solution, and again sublimed in a current of air which had been passed through wash bottles containing respectively a concentrated solution of potash and sulphuric acid. After having been sublimed in this manner about ten or twelve times, it was dissolved in re-distilled water and added to the sample of magnesian chloride. The whole was then filtered, evaporated to dryness, partly dehydrated, broken up and placed in a glass stoppered bottle. The usual tests were made as to its purity, but no traces of foreign matter were discovered.

The third sample of magnesian chloride, which was used for the final experiment in the last series, was at first treated in about the same way as the others. The precautions taken were somewhat greater, and the fractional precipitation with ammoniac oxalate was continued long after the last traces of calcium discoverable by the spectro-scope had disappeared from the precipitates of magnesian oxalate. The ammoniac magnesian chloride, already very pure, prepared from this sample, was then crystallised eight or ten times, the last six or eight re-crystallisations being conducted in platinum. From over a kilogram of magnesian chloride used in the beginning, the portion finally separated out consisted only of a few grms. This sample showed no traces of the sodium line when tested with the spectro-scope; indeed, several other samples, obtained from the mother liquors of the purest sample, gave equally satisfactory negative spectroscopic results. Since the magnesian chloride had contained in the first place a very noticeable amount of sodic chloride, the fact of the complete elimination of the impurity seemed a satisfactory indication of the elimination of other foreign materials. The double chloride was dried over an alcohol lamp, and treated in the same manner as the other samples.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, March 4th, 1897.

Mr. A. G. VERNON HARCOURT, President, in the Chair.

CERTIFICATES were read for the first time in favour of Messrs. Alaric Vincent Colpoys Fenby, B.Sc., Hutton Grammar School, Preston; R. Glode Guyer, 20, Queen's Road, St. John's Wood, N.W.; Tom Mitchell, Cemetery House, Shaw, near Oldham; Robert Howson Pickard, B.Sc., Southfield, Priory Road, Edgbaston, Birmingham.

Mr. CASSAL asked whether the officers had withdrawn the certificate of a candidate from the list to be balloted for that evening.

Professor THOMSON stated that the certificate of one of the candidates had been postponed pending further information.

Dr. ARMSTRONG remarked that such action had been taken by the officers on former occasions, and was within their discretion.

The following were duly elected Fellows of the Society:—Messrs. John Owen Alexander; Thomas Hannibal Aquino; William Arbuckle; John B. Ashworth; John Barclay, B.Sc.; Frank Bastow; Jyoti Bhushan Bhaduri, M.A.; James Herbert Brown; F. Hewlett Burton-Brown, M.A.; Alfred Cartmell; Masumi Chikashigé, B.Sc.; Alfred Foster Cholerton; Clarence Hamilton Creasey; James Crowther, B.Sc.; William Alfred Davis; William Diamond; John Wallis Dodgson, B.Sc.; Lawrence Dufy; William Buckland Edwards; Joseph Lake Gibbons; Alexander W. Gilbody, M.Sc., Ph.D.; Harold Walter Gough, B.A.; Ernest Goulding; Edward Graham Guest, M.A.; Vaughan Harley, M.D.; Ernald G. Justinian Hartley, B.A.; Thomas Hartley; Charles Heppenstall; John Holmes; Fred Ibbotson, B.Sc.; William Rose Innes, B.Sc., Ph.D.; David Smiles Jerdan, M.A., B.Sc.; Harold Johnson; Otis C. Johnson; Herbert King; William Robert Lang, B.Sc.; Theophilus Henry Lee; Charles Henry Martin; Barker North; Charles Henry Parker; Samuel Pollitt, B.Sc.; Herbert Spindler Pullar; William Ralston, B.Sc.; John Stewart Remington; Edward Rosling; Alfred Rutter; Frank Southerden; William James Stainer, B.A.; Henry Potter Stevens, B.A.; Leonard Sumner, B.Sc.; Harry Thompson; Andrew Turnbull, Ph.D.; Basil William Turner; Rustomji Navroji Umwalla; Samuel Matthew Walford; J. Wallace Walker, M.A., Ph.D.; and Meyer Wilderman, Ph.D.

In accordance with the Bye-law, the lists of the names of the Fellows recommended for election as official and ordinary Members of Council were read from the Chair.

Of the following papers those marked * were read:—

*34. "Some Hydrocarbons from American Petroleum. I. Normal and Iso-pentane." By SYDNEY YOUNG, D.Sc., F.R.S., and G. L. THOMAS, B.Sc.

The two pentanes were separated by fractional distillation from the "pentane" supplied by Merck of Darmstadt. This substance, which is obtained by the distillation of American petroleum, is a complex mixture of butanes, pentanes, and hexanes, with some benzene and a little hexanaphthene. A combination of a dephlegmator with a constant (or rather "regulated") temperature still-head was employed; the apparatus is fully described in the paper.

Some of the constants of isopentane were determined so as to compare them with those of the two specimens prepared synthetically; the agreement was found to be very satisfactory. The boiling-points under normal pressure are:—Isopentane, 27.95; normal pentane, 36.3. The specific gravities at 0° are:—Isopentane, 0.63930; normal pentane, 0.64539.

*35. "The Vapour Pressures, Specific Volumes, and Critical Constants of Normal Pentane; with a Note on the Critical Point." By SYDNEY YOUNG, D.Sc., F.R.S.

The critical temperature of normal pentane is 197.2, the critical pressure is 25,100 m.m., and the critical volume of a grm. 4.303 c.c. The vapour pressures and specific volumes were determined from low temperatures to the critical point, and the observations were taken as near to the critical point as possible (197.15), in order to obtain more complete experimental evidence regarding the condition of a substance at and very near that point.

The ratios of the absolute temperatures (boiling-points) and volumes to the critical constants, also the ratio of the actual to the theoretical density at the critical point (3.765), lead to the conclusion that, at the critical temperature and in the liquid state, the molecules are simple, like those of the gas.

DISCUSSION.

The PRESIDENT said that for many years he had been interested in the isopentane derived from American petroleum, having proposed that its flame should be used, under specified conditions, as a standard of light. He had purified it by shaking with sulphuric acid, and afterwards with soda. He had no doubt it was an improvement to use nitric acid, as Dr. Young had done. He had used a dephlegmator very similar to that described, but had never succeeded by fractional distillation in obtaining a substance of such a constant boiling-point as that obtained by Dr. Young. He was much interested to hear this proof by Dr. Young of the identity of isopentane with that which is obtained from amyl iodide, as experiments on the illuminating power did not quite settle this question.

Dr. ARMSTRONG thought that it was not quite certain that the isopentane used by Dr. Young was a single thing, and it was probably very difficult to obtain satisfactory proof of purity. In the case of the specimen derived from amyl alcohol, he considered it likely that this was a mixture of two hydrocarbons, as it did not appear that the amyl alcohol of fusel-oil had first been separated into its two constituents.

Dr. CRAW said that Dr. Young seemed to have shown that the critical temperature could be determined accurately for the class of compounds on which he had worked; but he asked whether the critical temperature is capable of being determined with the same accuracy for all classes of compounds.

Mr. GROVES agreed with Dr. Armstrong as to the necessity of taking special precautions in purifying materials and of not trusting to those supplied by manufacturers.

Dr. THORPE said he had had some experience in the preparation of isopentane. He had obtained it from amyl alcohols of very different origin. The question was whether the isopentane derived from fusel oils of very different origin would give the same hydrocarbon. He had found that all the specimens of isopentane gave practically the same boiling-point and density numbers as those obtained by Dr. Young. Nevertheless, Mr. Rodger and he had found that the viscosities of isopentane derived from different sources varied considerably, and it was significant that their specific volumes and specific gravities agreed closely with those recorded by Dr. Young. In the case of other liquids differently prepared and purified, the same viscosity numbers were obtained: for example, Prof. Dunstan had provided him with a sample of pure ether, and Dr. Perkin also provided him with a sample, and the viscosities of the two were in perfect agreement. Similarly, he had compared two samples of benzene of different origin, and here also the two samples gave the same viscosity number. On this account he was inclined to question the homogeneity of isopentane prepared from amyl alcohol.

Dr. YOUNG, in reply said he had spent many months in purifying the materials, and was satisfied that they were

pure. He was, indeed, surprised that doubt should be cast upon the matter.

In the fractionation of the pentane and isopentane, he started with considerable quantities of materials, about 1500 grms., and he obtained about 110 grms. of pure substance. The liquid was fractionated about twenty times, and the loss was nearly 20 grms. in each fractionation. There were only, as far as he knew, three isomeric pentanes, and, as they had distinctly different boiling-points, he did not see that there could be any mistake as to their identity. The critical temperatures and pressures are very delicate tests of the purity of the substance. With slightly impure ether, the difference in critical pressure is very considerable.

With regard to the fractionation of pentane and isopentane, the ordinary methods of fractional distillation would not answer. With the ordinary still-head, the loss by evaporation was greater than the gain by distillation, but the regulated still-head he had used gave most satisfactory results. He had no hesitation in saying that, with all substances which do not undergo decomposition when heated, the critical point can be determined within one-tenth of a degree.

*36. "On the Freezing-point Curves of Alloys containing Zinc." By C. T. HEYCOCK, F.R.S., and F. H. NEVILLE.

The paper is divided into two sections, the first dealing with cases where the freezing-point of zinc is depressed by the addition of another metal, the second with the cases where it is raised by such an addition.

In Section I. complete binary alloys of the following pairs of metals, zinc-cadmium, zinc-aluminium, zinc-tin, and zinc-bismuth, are given. In the first three cases the metals appear to be miscible with each other in all proportions; but for zinc-bismuth the freezing-point curve shows the horizontal line of identical freezing-points characteristic of the state when the alloy has separated into two conjugate liquids.

Taking the freezing-point of zinc as 419°, the authors find for the temperatures of freezing and the composition of the eutectic mixtures the following:—

Alloy.	F.P.	Atomic p.c. of zinc.
Zn—Cd ..	264.5	74
Zn—Al ..	281	11
Zn—Sn ..	198	84
Zn—Bi ..	254.5	91.8

They do not consider that there is any indication of these metals combining chemically to form definite compounds when they are melted together.

Dilute solutions of the following metals in zinc were also examined:—Lead, thallium, antimony, magnesium.

The authors find that, with the exception of aluminium and cadmium, all the above-mentioned metals, when added in small quantities to molten zinc, cause the same atomic fall; that is, an alloy containing 1 atomic weight in solution in 99 atomic weights of zinc has a freezing-point lower by 5.1° than that of pure zinc. Cadmium causes a somewhat smaller and aluminium a decidedly smaller depression. The atomic depression of 5.1°, when used with van't Hoff's equation for the latent heat, gives 28.3 calories for the latent heat of fusion of zinc, instead of Person's value of 28.13.

In Section II. dilute solutions of copper and of gold in zinc are considered, and also a complete freezing-point curve for all alloys of zinc and silver.

When small quantities of any one of these three metals are added to molten zinc the effect is the same: the freezing-point is raised, and the whole mass of metal appears to solidify at a temperature above the freezing-point of pure zinc. The rise in the freezing-point, moreover, is proportional to the amount of the second metal present. But when as much as 2 atomic per cents of silver or of copper, or 3.3 of gold, have been added, the phenomenon alters, apparently abruptly. There are now

two freezing-points, a higher one which is very fugitive, and which is followed by the precipitation of much solid, and a lower one which is very constant, and which is independent of the amount of the added metal so long as the above-mentioned minimum is present. In the zinc-silver curve singularities are also found near 70 and 60 atomic per cents of zinc, indicating the existence of more or less stable compounds; but the formula of these compounds is uncertain. Near 37.5 atomic per cents of zinc there is another well-marked angle and a series of eutectic second freezing-points, the phenomenon being possibly due to the separations of the alloy into conjugate liquids.

The authors also describe briefly some of the physical properties of the zinc-silver alloys, which appear to change in character at the angles of the curve.

The composition of the silver-zinc alloys at each freezing-point was determined by extracting a portion in the liquid state, and estimating the percentage of silver by a volumetric analysis.

DISCUSSION.

Mr. NEWLANDS asked whether the formulæ of the compounds which were supposed to exist at certain points,—viz., AgZn , AgZn_2 , and AgZn_3 ,—represented atoms of the metals, or only that the metals were present in that proportion.

Mr. GROVES asked whether there was any connection between the colour of the silver-zinc alloy and any of the points on the curve.

Mr. JENKINS asked whether the colour of the alloy was in any way due to the effect of mechanical stress during sudden cooling.

Dr. CRAW asked whether the atoms in the alloy were in the monatomic state, and whether the alloy might be compared with a solution.

Mr. NEVILLE, in reply, said the formulæ were only empirical. They were disposed to think that the dissolved metal was in a monatomic state. The colour of the alloy did not seem to have any connection with the points on the curve, and, although great mechanical stress occurred in its production, this did not seem to afford an explanation of the colour.

*37. "*The Oxides of Cobalt and the Cobaltites.*" By ARTHUR H. MCCONNELL and EDGAR S. HANES,

The authors describe a method for the preparation of alkali cobaltites, and show that cobalt forms an oxide, CoO_2 , and an acid, H_2CoO_3 , which have hitherto been looked upon as hypothetical, and a series of alkali salts on the type of potassium cobaltite, K_2OCoO_2 . The conclusions the authors arrive at are as follows:—

1. That Durrant (*Proc.*, 1896, xii., 96, 244) has not produced sufficient evidence for the existence of either cobaltic acid or cobalt percarbonate.

2. That cobaltous acid corresponds with cobalt dioxide, and forms alkali salts fairly stable in solution, which solutions have an unmistakable green colour.

3. That cobalt forms a series of compounds with other metals in which the cobalt is part of the acid radicle.

4. That cobaltous acid and cobaltites are strictly analogous to manganous acid and manganites, thus showing that the properties of cobalt are closely allied to those of the other elements associated with it in the periodic classification. Manganese is readily oxidised to the peroxide MnO_2 , but cobalt much less readily yields the corresponding peroxide CoO_2 .

5. In view of the fact that cobalt dioxide does undoubtedly exist in a number of compounds, the authors suggest that the oxides of cobalt should be re-named, to bring them into line with the corresponding oxides of manganese.

6. It is highly improbable that the formation of this green solution will prove to be of any use for the separation of cobalt from nickel, either quantitatively or qualitatively.

*38. "*A New Synthesis in the Sugar Group.*" By HENRY J. HORSTMAN FENTON, M.A.

In previous communications it has been shown that the acid (dihydroxymaleic acid) obtained by oxidation of tartaric acid in presence of iron decomposes, on heating with water, almost quantitatively into glycollic aldehyde and carbon dioxide. Also, that this aldehyde, when heated in a vacuum, undergoes condensation, yielding a sweet-tasting, solid gum, which has the formula $\text{C}_6\text{H}_{12}\text{O}_6$.

The present paper describes an investigation which has been made upon the properties of this condensation product.

It is easily soluble in water, and its solution quickly reduces Fehling's solution and ammoniacal silver nitrate. It gives various colour reactions characteristic of "sugars," and, after purification with alcohol, yields, with phenylhydrazine, a normal hexosazone, $\text{C}_{18}\text{H}_{22}\text{N}_4\text{O}_4$, melting at $168-170^\circ$. Heated with water to 140° , it yields furfural. It is optically inactive, and appears to be incapable of fermentation by yeast.

The purified "sugar," when further heated in a vacuum to $100-106^\circ$, loses water and becomes hard and brittle. After two to four hours' heating it has the composition $\text{C}_{12}\text{H}_{22}\text{O}_{11}$, and after twenty-four hours' heating the composition nearly approximates to $\text{C}_6\text{H}_{10}\text{O}_5$.

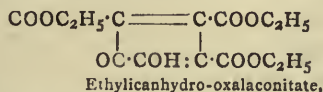
The conditions under which tartaric acid may be converted into dihydroxymaleic acid by atmospheric oxygen exhibit close analogies with some of the essential conditions of vegetable growth; and it is suggested that the direct production of a "sugar" in the manner above indicated may possibly help to throw light upon the natural formation of carbohydrates.

39. "*The Dinitrosamines of Ethylene Aniline, the Ethylene Toluidines and their Derivatives.*" By FRANCIS E. FRANCIS, Ph.D., B.Sc.

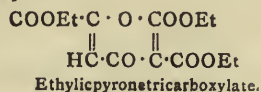
The dinitrosamine of ethylene aniline gives *p*-dinitroso-ethylene aniline hydrochloride on treatment with a mixture of glacial acetic and hydrochloric acids, and the resulting tetramine yields quinone on oxidation, showing that it is a *p*-diamine, ethylene-*p*-phenylene diamine. The dinitrosamines of ethylene-*o*-toluidine and ethylene-*m*-toluidine yield di-nitroso compounds, which on reduction pass into corresponding tetramines, whereas the dinitrosamine of ethylene-*p*-toluidine is decomposed. This clearly shows that in the substances investigated the nitroso-group can only pass under the treatment described to the *p*-position in the benzene nucleus.

40. "*Contribution to the Knowledge of the β -Ketonic Acids.*" Part V. By S. RUHEMANN, M.A., Ph.D., and A. S. HEMMY, B.A., M.Sc.

Whilst studying the interaction between the sodium derivative of ethylic oxalacetate and ethylic chlorofumarate, the authors observed the formation of two isomerides of the formula $\text{C}_{14}\text{H}_{16}\text{O}_8$. One of them, melting with decomposition at 200° , forms blue salts with alkalis which, by an excess of the reagent, become colourless. The other, which melts at 123° , does not yield coloured salts, but gives a dark red colouration with ferric chloride. The authors arrive at the view that in the formation of these compounds, ethylic oxalacetate alone takes part, and they represent the constitution of the substance decomposing at 200° by the formula—



the other by the symbol—



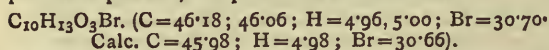
41. "Enantiomorphic Forms of Ethylpropylpiperidonium Iodide." By CLARE DE BRERETON EVANS.

It has been found that ethylpropylpiperidonium iodide ($C_5H_{10}EtPrNI$) may be obtained from its solution in absolute alcohol, in right-handed and left-handed crystals. The enantiomorphism, however, is of a purely crystallogenic order, due to the arrangement of the molecules in the crystal, and not to the position of the atoms in space. This is proved by the optical inactivity of the substance in solution, as well as by the fact that either variety may be converted into the other by recrystallisation, the behaviour being like that observed in the case of sodium chlorate and bromate, &c., &c.

42. "Further Note on Ketopinic Acid — Pinophanic Acid." By W. S. GILLES and F. F. RENWICK.

The further investigation of the acid obtained by oxidising the solid hydrochloride from pinene with the strongest nitric acid (*Trans.*, 1896, lxi., 1397) has brought to light a variety of interesting points.

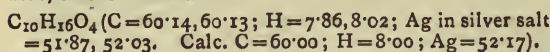
Although bromine alone does not attack it, ketopinic acid is readily brominated if a small quantity of phosphorus be present; the product has the formula—



Monobromoketopinic acid melts at 181° ; it is readily soluble in ether, acetone, acetic acid, and ethylic acetate, but sparingly so in benzene, chloroform, and hot water. When heated with aniline or quinoline, it is re-converted into ketopinic acid.

The hydroxime of ketopinic acid is merely hydrolysed when boiled with 50 per cent sulphuric acid, being re-converted into ketopinic acid.

When cautiously fused with caustic soda, or even when boiled with an alcoholic solution of sodium ethylate, ketopinic acid is converted into a dibasic acid, *pinophanic acid*, of the formula—



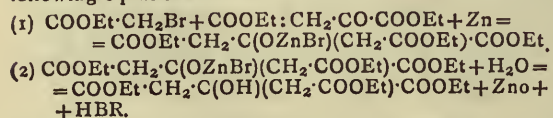
Pinophanic acid melts at 203° ; it is insoluble in benzene, light petroleum, and chloroform, moderately soluble in hot water and hot ethylic acetate, and readily soluble in alcohol, acetone, and ether. Like ketopinic acid, it does not combine with bromine.

Although but slowly attacked, ketopinic acid is oxidised by prolonged digestion with neutral potassium permanganate solution. The product appears to be identical in composition with Kipping's camphotricarboxylic acid and Marsh and Gardner's camphoic acid, but more closely resembles the latter.

These acids are now being fully investigated.

43. "A Synthesis of Citric Acid." By W. T. LAURENCE, B.A., Ph.D.

Ethylic citrate was obtained synthetically by the condensation of ethylic bromacetate with ethylic oxalylacetate in the presence of zinc, as indicated by the following equations:—



The yield of ethylic citrate is very poor, owing to other reactions proceeding simultaneously. To further confirm the formation of ethylic citrate, it was converted into the calcium salt of citric acid, and a substance obtained showing the characteristic properties of calcium citrate. The same salt was also obtained by heating the zinc compound formed in equation No. 1 with alcoholic potash and precipitating the calcium citrate from the hot solution.

The results were all confirmed by analysis. The above synthesis of citric acid seems to be of interest as being

more direct and simple than the synthesis from sym-dichloroacetone or from ethyl- γ -cyanacetate.

"Ordinary Meeting, March 18th, 1897.

Mr. A. G. VERNON HARCOURT, President, in the Chair.

MESSRS. H. P. Stevens, J. W. Walker, W. Arbuckle, N. T. N. Wilshire, M. Wildermann, W. J. Pope, A. W. Crossley, H. R. Le Sueur, J. H. Miller, R. D. Littlefield, F. H. Neville, W. M. Heller, G. McGowan, C. M. Crossman, J. Holmes, and F. Southerden were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Alfred Hunter Boylau, Eilerslie, Richmond Road, Ealing, W.; Henry Norris Davidge, 37, Duke Street, Grosvenor Square, W.; Charles Henry Field, The Elms, Green Street Green, Orpington; Thomas Girtin, B.A., 125A, Highbury New Park, N.; James Jones, 117, Old Christchurch Road, Bournemouth; Charles MacCulloch, 395, Collins Street, Melbourne; George Fowlie Merson, 65, Northumberland Street, Newcastle; Thomas Tickle, 4, Pakenham street, W.C.

In accordance with the By-laws, the following communication was read from the Chair:—

We, the undersigned, beg to propose Prof. William Ramsay, Ph.D., F.R.S., as President of the Chemical Society in succession to Mr. A. Vernon Harcourt, M.A., M.D., D.C.L., F.R.S.:—

Baly, E. C. C.; Baker, C. F.; Burgess, Herbert E.; Blount, Bertram; Blundstone, Edward, R.; Berncastle, Richard; Cassal, Charles E.; Chattaway, F. D.; Chapman, Alfred C.; Collie, J. Norman; Chorley, John C.; Coste, J. H.; Crossley, Arthur; Dufton, S. T.; Ekins, Arthur E.; Edwards, W. Buckland; Evans, R. C. T.; Elborne, William; Earle, Alfred; Elford, P.; Forster, M. O.; Graham, Edward; Grimwood, R.; Harley, Vaughan; Harvey, Sydney; Baker, Julian L.; Floris, R. B.; Jackman, E. J.; Jones, Cecil; Johnson, David; Kipping, Stanley F.; Kellas, Alex. M.; Kingzett, C. T.; Ling, A.; Lapworth, A.; Livingston, W. T.; Littlefield, R. D.; Routledge, R.; Lamb, Edmund; Moody, Gerald T.; Bodmer, R.; Moor, C. G.; Millar, J. H.; McCrae, J.; Macdonald, G.; Mills, Charles; McGowan, Geo.; Pope, W. J.; Parry, Ernest J.; Pidon, Harold; Plimpton, R. T.; Richmond, H. Droop; Simpson, Arthur M.; Stevens, H. P.; Travers, M. W.; Le Sueur, H. R.; Wade, John; Wagner, W. G.; Wilshire, N. T. M.; Walker, J. Wallace; Sworn, Sydney A.; Cornish, Vaughan; Sykes, Walter J.; Waterhouse, Robert; Chattaway, William; Priest, Martin; Marsh, J. E.; Muspratt, Edmund K.; Mitchell, C. A.; Muspratt, Sydney K.; Adams, Arthur; Marshall, Arthur; Wilson, John; Bone, W. A.; Fisher, E. H.; Chapman, Arthur J.; Wheelwright, E. W.; Lewis, W. H.; Sudborough, J. J.; Veley, V. H.; Walker, James; Colman, Harold G.; Christopher, George; Macnair, D. S.; Bell, Chichester A.; Wertheimer, J.; Hodgkin, John; Archbutt, Leonard; Muir, M. M. Pattison; Shaw, G. E.; Taylor, R. L.; Colwell, J. Kear; Cribb, Cecil H.; Butterfield, W. J. Atkinson; Hanes, Edgar S.; Sutherland, D. A.; Teed, Frank L.; Fulcher, L. W.; Heller, W. M.; Sandford, P. Gerald; Snape, H. Lloyd; Knight, J. B.; Cooper, A. J.; Adams, P. T.; Adams, M. A.; Eiloart, A.; Corfield, W. H.; Muter, J.; Muter, A. H. M.; Dodd, W. H.; Koningh, L. de; Mawer, W. F.; Lascelles, P. B.; Bruce, James; Turpin, G. S.

Professor COLLIE stated that the nomination of Professor Ramsay had been made without his knowledge or his permission.

Of the following papers those marked * were read:—

"44. "On the Atomic Weight of Carbon." By ALEXANDER SCOTT, M.A., D.Sc.

The object of this paper is to call attention to the unsatisfactory nature of the experimental evidence on which the determinations of the atomic weight of carbon rest. The two methods on which reliance is chiefly placed are shown to be only in agreement because a source of error which affects both acts in opposite directions. This is due to an erroneous determination of Dumas and Stas, in 1840, of the expansion produced in potash solutions by the absorption of carbon dioxide. All later workers at this problem seem to have accepted their conclusions without further verification. The seriousness of this source of error is apparent when we consider that over 40 m.grms. is the correction for the weight *in vacuo* of the carbon dioxide in one experiment alone. The correction per grm. of carbon dioxide is shown to be 0.56 to 0.57 c.c., instead of 0.15 c.c. as taken by Dumas and Stas. The probable effect of the gases "occluded" in the copper oxide is next considered, and, as far as possible, allowed for. It is shown by many determinations that the experiments of Richards give the quantity as much too great, the mean result of his best experiments being 0.088 per cent by weight of nitrogen in copper oxide made from the nitrate, whilst the experiments here described give as a mean only about 0.007 per cent.

Other sources of error and the best methods of making more accurate determinations are next considered. The re-calculated values are 12.008 from the combustion of the various forms of carbon, and 12.050 from the conversion of the monoxide into the dioxide.

DISCUSSION.

In reply to questions from Mr. GROVES, Mr. BLOUNT, Mr. HEYCOCK, and the PRESIDENT, Mr. SCOTT stated that 150 grms. of the oxalate furnished about 4 grms. of carbon. In the case of the potash solution, it was the expansion of the liquid which had to be corrected for. The rate of absorption of carbon dioxide by potash solution at any given time was affected by the amount of carbon dioxide which had been already absorbed. Carbon monoxide was completely absorbed by potash after some time.

"45. "On a New Series of Mixed Sulphates of the Vitriol Group." By ALEXANDER SCOTT, M.A., D.Sc.

This paper describes a new series of mixed sulphates of the form $(M,N)^nSO_4 \cdot H_2O$. The most interesting is the ferrous cupric sulphate, the colour of which is reddish brown; it dissolves in water, giving a blue green solution.

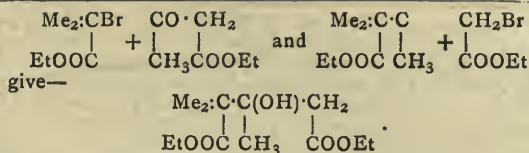
The composition of this salt on analysis corresponds to the formula $(CuFe)SO_4 \cdot H_2O$, or $(Fe_5Cu_2)(SO_4)_{7.7}H_2O$. These salts are made by adding about an equal bulk of strong sulphuric acid to solutions of the mixed sulphates.

DISCUSSION.

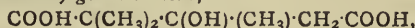
Mr. SPILLER, referring to investigations he had conducted on some double sulphates of this group, crystallised from water, the results of which were communicated to the British Association ten years ago, said that he was led to the conclusion that the amount of water of crystallisation in such mixed sulphates was the mean of that present in their constituent salts.

"46. "A Synthesis of Camphoronic Acid." By WILLIAM HENRY PERKIN, jun., F.R.S., and JOCELYN FIELD THORPE, Ph.D.

In a previous communication (*Proc.*, 1896, xii., 155) experiments were described dealing with the action of metallic zinc on mixtures of bromo-ethylic salts and ketones or ketonic acids; notably on mixtures of ethylic aceto-acetate and ethylic α -brom-iso-butyrate, and of ethylic dimethylacetoacetate and ethylic bromoacetate, and it was shown that the same hydroxy-ethylic salt, namely, ethylic- β -hydroxy- α - β -trimethyl glutarate was in each case produced thus:—

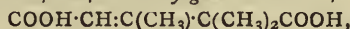


This ethylic- β -hydroxy- α - β -trimethylglutarate (b. p. 165°, 30 m.m.), as previously stated, splits up, on hydrolysis with alcoholic potash, into acetic and isobutyric acids. When, however, it is boiled with dilute hydrochloric acid, it does not behave in this way, but yields considerable quantities of the corresponding β -hydroxy- α - β -trimethylglutaric acid,—

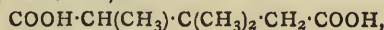


which is a crystalline body, separating from a mixture of light petroleum and ethylic acetate in glistening prisms melting at 128°.

In the previous communication, an acid melting at 148°, obtained by the action of alcoholic potassium cyanide on ethylic- β -brom- α - β -trimethyl glutarate, was described as a trimethylglutaric acid: we now wish to correct this statement. Alcoholic potassium cyanide is apparently without action upon the bromethylic salt at the temperature of the boiling water-bath, and, on hydrolysing the product with alcoholic potassium hydroxide, the unsaturated acid, α - β -trimethylglutaconic acid,—

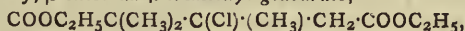


melting at 148°, is produced, and not the trimethylglutaric acid as was at first supposed. This acid is remarkably stable, and is not affected by boiling with sodium amalgam. When, however, its boiling solution in alcohol is treated with sodium, the unsaturated acid is gradually reduced to α - β -trimethylglutaric acid, $\text{COOH} \cdot \text{CH}_2 \cdot \text{CH}(\text{CH}_3) \cdot \text{C}(\text{CH}_3)_2 \cdot \text{COOH}$, which crystallises from dilute hydrochloric acid in prismatic needles melting at 109°; the anhydride of this acid melts at 38°, and yields, on treatment with aniline, an anilic acid forming lustrous plates from dilute alcohol melting at 155°. Although the melting-points of the acid and of the anil are very similar to those of the trimethylglutaric acid which Balbiano obtained from camphoric acid, it does not appear that the acids are identical, and it is probable that Balbiano's acid is the isomeric α - β -trimethylglutaric acid,—



as this chemist suggests.

On treating ethylic β -hydroxy- α - β -trimethylglutarate with phosphorus pentachloride, the chlorethylic salt, namely, β -chlor- α - β -trimethyl glutarate,—



is obtained as a colourless mobile liquid which boils at 139° (20 m.m.). When this substance or the corresponding bromo-derivative is heated with alcoholic potassium cyanide in a closed tube at 160° ethylic- β -cyano- α - β -trimethyl glutarate,—



is obtained after twelve hours as an oily liquid boiling at 180—185° (25 m.m.). It is difficult to isolate in the pure condition owing to the presence of varying quantities of the ethylic salt of trimethylglutaconic acid, a substance which boils at about the same temperature as the nitrile (175°, 30 m.m.).

This nitrile was hydrolysed by boiling with dilute hydrochloric acid, and after filtering from the trimethylglutaconic acid, which separated on cooling, the filtrate yielded on neutralisation with ammonia and addition of barium chloride no precipitate, but on boiling, a quantity of a sparingly soluble barium salt separated. This salt was collected, well washed, and decomposed by boiling with the calculated quantity of sulphuric acid; the filtrate from the barium sulphate was then evaporated to a small

bulk, when on cooling a crystalline acid separated, which melted at 157° with decomposition, and on analysis gave the following numbers:—

0.1258 grm. gave 0.2276 grm. CO_2 and 0.0746 grm. H_2O . Calc. for $(\text{C}_9\text{H}_{14}\text{O}_6)$: $\text{H}=6.40$; $\text{C}=49.50$. Found: $\text{H}=6.58$; $\text{C}=49.34$. α - β -Trimethyltricarballic acid, $\text{COOH}\cdot\text{CH}_2\text{C}(\text{COOH})(\text{Me})\text{C}(\text{Me}_2)\text{COOH}$.

That this acid is identical with camphoronic acid is, in our opinion, proved by the following considerations:—

(1) The synthetical acid gives the same results on analysis, and melts at the same temperature as camphoronic acid.

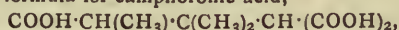
(2) When equal quantities of the synthetical acid and camphoronic acid are intimately mixed, the mixture melts at exactly the same temperature, i.e., 157°, with decomposition.

(3) It gives, when dissolved in a slight excess of ammonia, no precipitate with barium chloride until the liquid is warmed, and then the insoluble barium salt separates exactly as in the case of camphoronic acid.

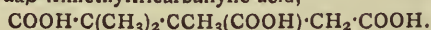
(4) When heated with acetyl chloride, both the acids are converted into an anhydro-acid, which melts in both cases at 135–136°.

The anhydrocamphoronic acid from the synthetical acid gave on analysis the following results:—Found, $\text{C}=54.03$; $\text{H}=6.11$. $\text{C}_9\text{H}_{12}\text{O}_5$ Calc. $\text{C}=54.00$; $\text{H}=6.00$ per cent.

In a previous communication (*Proc.*, 1896, xii., 192),* one of us had occasion to express the opinion that Tiemann's formula for camphoronic acid,—



which contains the group $-\text{CH}(\text{COOH})_2$ must be incorrect, on account of the fact that camphoronic acid, when heated with water at 230°, is not decomposed with elimination of CO_2 . The experiments which we have briefly described in this communication appear to us to prove that camphoronic acid has the constitution first proposed by Bredt (*Ber.*, 1893, xxvi., 3048), namely, that of an $\alpha\alpha\beta$ -trimethyltricarballic acid,—



(To be continued).

PHYSICAL SOCIETY.

Ordinary Meeting, March 26th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

At the invitation of Dr. S. P. Thompson, the meeting was held at the Technical College, Leonard Street, Finsbury.

Mr. ROLLO APPLEYARD read a paper on "*Liquid Coharers and Mobile Conductors*," and showed the following experiments:—

(1.) A glass tube, containing mercury and paraffin oil, is shaken up until the mercury divides into small spheroids. The resistance of the chain of spheroids under these conditions is several megohms. Coherence can be brought about by a direct current, a spark, or by a Hertz oscillator. The coherence is visible, the spheroids forming into large globules. At the same time, the resistance falls to a fraction of an ohm. (2.) An unstable emulsion is formed by shaking water and paraffin oil together, in a glass tube, called by the author a "rain" tube. The oil may be coloured with alkanet root. By electrification, the water suspended in the oil is suddenly precipitated in a shower through the oil, precisely as rain is precipitated in the air, after thunder. (3.) A mixture of paraffin oil and water is poured into a photographic

dish, just covering the bottom, and a little mercury is poured in. Any two separate globules of mercury in the dish are then connected by wires to a battery of about 200 volts, through a reversing-key. A momentary tap of the key causes instantaneous deformation of the mercury, especially of the globule connected to the negative pole. If the current is kept on, the negative globule sends forth a long tentacle of mercury across the dish to the positive globule. The tentacle may break into spheroids. Intermediate globules send forth "fingers" towards the positive terminal-globule, and, by continued application of the current, the "fingers" link intermediate globules,—illustrating the nature of liquid coherence. By using the current-reverser as a telegraphic transmitting-key, the motions, to right or left, of the "finger" of any stray globule, may be interpreted to form the letters of the Morse code. By a succession of taps of the key in one direction or the other, a globule can be made to "caterpillar" along the dish.

Prof. RAMSAY said he had once attempted to facilitate churning by the application of 8 or 9 volts to some milk. He thought the cream came a little faster, but it turned sour very quickly.

Prof. FITZGERALD thought that the effects observed in experiment 3 were the result of current, and not of electrostatic changes; and he would like to know the value of the actual current used. There was no doubt that the motions were due to variations in capillarity.

Mr. SHELFORD BIDWELL asked how the mercury was formed into spheroids in the tube in experiment 1.

Mr. APPLEYARD, in reply to Prof. Fitzgerald, said it was not easy to define the circuit, as the terminal-globules were rather capricious, but he would try and measure the current in some particular case. The mercury-tube in experiment 1 was shaken in a horizontal plane; the operation took about ten minutes. Equal volumes of mercury and oil was a good proportion. One quarter of the length of the tube should be left as an air-space.

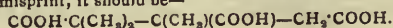
Prof. DALBY then exhibited five pieces of apparatus:—(1.) A Kinematic Slide. (2.) An Inertia Apparatus with trifilar suspension. (3.) A Wilberforce Spring. (4.) An Ewing's Reading-telescope. (5.) A Kinematic Hooke-gauge. Models 1, 2, 4 and 5 illustrated the various degrees of freedom of bodies restrained at different numbers of points. It was shown with 3 that in extending a spiral spring there results a certain amount of twisting. If a mass is hung at the lower end of the spiral in such a way that, when suddenly released after extension of the spring, the time of oscillation of the mass in the horizontal plane (rotation) is the same as the time of vertical oscillation, then the tendency to twist results in a change of energy which alternates between the rotary and linear forms.

Mr. BOYS drew attention to the conditions of restraint, and suggested a criterion for determining whether a piece of mechanism was designed for minimum strain on the structure: a thin wedge slipped under any one point of contact should not disturb the other points of restraint.

Prof. FITZGERALD pointed out the effect of symmetry upon the motion of the spring of 5. The spiral happened to be an unsymmetrical form; the change of phase from vertical to rotary oscillation was therefore rapid. In the case of the vibration of a symmetrical stretched cord the change of phase would be very slow.

Dr. THOMPSON exhibited two Kinematic Models depending upon the principle that any simple harmonic motion may be considered as the resultant of two oppositely directed motions. The first illustrates the synthesis of two opposite circular motions of equal period and magnitude to form a straight line motion; the second shows the combination of two simple harmonic motions of equal period and amplitude in any difference of phase, to form a circular motion. In each case the motion is communicated to a stylus by a link-gear, operated by two wheels rotating in opposite directions. In the first appa-

* The formula given here from Bredt's constitution of camphoronic acid is a misprint; it should be—



ratus the wheels are pivoted about their centres, and the link-gear is pinned to one point on the flat surface of each wheel, near the circumference; in the second apparatus the wheels rotate as eccentrics at 180° to one another, and the motion to the link-gear is communicated by thrust-rods, held by springs against the peripheries of the corresponding wheels.

Dr. THOMPSON further exhibited a device for projecting, by lantern, the rotating magnet and copper disc of Arago. The curious rotations and lateral movements of iron filings, in a revolving magnetic field, were similarly projected on a screen. He also showed some experiments with a heat-indicating paint, made from a double iodide of copper and mercury, discovered twenty years ago by a German physicist. At ordinary temperatures the paint is red, but at 97° it turns black. If paper is covered with this substance, and then warmed at a stove, the change is effected in a few seconds. Various designs can be wrought upon the back of the paper in dead-black or gold, so that when warmed they appear in red or black on the front, according to their respective absorptive powers. Or local cooling by the hand will yield a silhouette. If the paper is allowed to cool, the silhouette vanishes, but it appears again when the paper is re-heated. It has thus a kind of thermal "memory." A yellow double iodide of silver and mercury is even more sensitive. It changes from yellow to dark red at 45° C.

Lastly, Dr. THOMPSON exhibited a Kinematic Model of Hertz-wave Transmission. A row of lead bullets is suspended from strings, so that the bullets hang clear of one another by about an inch, in a right line. The strings are meshed, and herein the model differs from the well-known wave-models used in acoustics. If the attempt is made to send an acoustic form of wave through the system, by giving an impulse to the first bullet in the plane of the other pendulums, it fails immediately, owing to the slackening of parts of the meshes. Thus only transverse vibrations can be transmitted. To illustrate the propagation of a Hertz-wave, a heavy pendulum, oscillating in a plane at right angles to the line of bullets, at one end, represents the Hertz "oscillator." A metal ring, mounted horizontally on a trifilar suspension, and properly "tuned," represents, at the distant end, the Hertz "resonator." Waves, formed by the transverse vibrations of successive bullets, are then propagated from end to end.

Prof. FITZGERALD said the model was specially interesting as illustrating the difference in velocities of propagation of a given wave, and of the energy corresponding to it. The model did not accurately compare with ether, because in ether the rate at which the energy is propagated is the same as that of the wave. The difference of the two rates, for any medium, depended upon the "dispersion" of the medium. By slight alteration of the pendular-suspensions this dispersion might be made different at different parts of the model, and would then correspond to certain known cases of "anomalous" dispersion. Or again, it might be made to illustrate the theory of Helmholtz with regard to the vibrations of the molecules of glass; according to which, the vibration of the molecules alters the vibrations of the waves, so that dispersion occurs, and the energy is not propagated at the same rate as the waves themselves. It was shown by Michaelson that it was possible to have a medium in which the energy is propagated in one direction and the wave in another. This was attained, in a magnetic model, by Ewing. The mesh apparatus indicated how a model could be made which should give our "harmonics" and "over-tones" very different from one another; where different wave-lengths would be propagated with different velocities, and the over-tones would correspond to the differences. Further, it indicated a mechanism for producing any desired spectrum, such, for instance, as that of hydrogen. A somewhat similar model had been designed by Glazebrook for illustrating the absorption-bands of a medium when the rate of vibration was the same as

the free period of the vibrations of each of the molecules, which is the theory of Helmholtz, but it was not such a simple model. The experiment of red paper changing to black was interesting as illustrating a red spectrum varying with temperature.

Mr. SHELFORD BIDWELL proposed a vote of thanks to all the exhibitors, and the Society adjourned until April 9th.

NOTICES OF BOOKS.

Principles and Practice of Agricultural Analysis: a Manual for the Examination of Soils, Fertilisers, and Agricultural Products. For the Use of Analysts, Teachers, and Students of Agricultural Chemistry. Vol. III. — Agricultural Products. By HARRY W. WILEY, Chemist to the U.S. Department of Agriculture. Easton, Pennsylvania: Chemical Publishing Co. 1897. Pp. 644.

THIS volume opens with a carefully compiled dissertation on sampling, drying, incineration, and extraction—subjects in which it is possible, even for experienced practitioners, to go astray, with grievous inconvenience and injury to their professional reputation.

As regards the scope of the work, we note that whilst hops are included, opium with of course morphia are left outside the pale. Nor are organic matters, odoriferous and insubstantial, admitted, except as regards the pigments of wines. The importance and the delicacy of the procedures for desiccation and incineration are certainly not exaggerated. The apparatus for dyeing samples *in vacuo* or in currents of inert gases, though somewhat complicated, will be found of great utility.

Part II. is devoted to sugars and starches. In considering the areometric method for the determination of sugars, we regret to find the total omission of Twaddle's instrument and scale in favour of Baumé.

Part III. treats of the separation and determination of carbohydrates in agricultural products, crude and manufactured; and Part IV. deals with fats and oils. Here the usual analytical methods are expounded. It is truthfully admitted that the spectroscopy is of little practical utility in oil analysis.

In the fifth, sixth, and seventh parts, we find an account respectively of the separation and determination of nitrogenous bodies, of dairy produce, and of miscellaneous agricultural products. Here the defects of ensilage are admitted. The writer condemns as fraudulent the sale of horse-flesh as beef and pork, but he rightly holds that it is unobjectionable if sold under its own name.

The author's orthography is apt to set the teeth of us Britishers on edge, and raises the question why, if Americans have gone so far in the "fonetic" direction, they do not fully adopt the code of the spelling reformers?

Concerning the value of Mr. Wiley's work as a whole, there can be no difference of opinion.

Chapters on the Aims and Practice of Teaching. Edited by FREDERICK SPENCER, M.A., Ph.D., Professor of the French Language of Literature at the University College of North Wales; formerly Chief Master on the Modern Side at the Leys Schools, Cambridge. Cambridge: University Press. 1897. 8vo., pp. 284.

MUCH of this book deals with subjects on which our opinion is little more valuable than that of the "man in the street." At the same time, whilst admitting the wealth of thought which is here put forth, we must presume to express our regret at the predominance of "verbalism" of some of the writers. Too often they

seem to forget the glaring facts that so long as boys are compared by the figure which they make in classical subjects the gravest mistakes are committed. Every one knows that Justus Liebig and Charles Darwin were in their school-days set down as hopeless dunces. But when released from verbalism they became, each in his department, great creative spirits, of more value to the world than generations of grammarians. Yet one of the writers here before us "has no hesitation in saying that, on the average, boys trained on the classical side of our public schools make better men of science and medicine than the boys who come to the University from the modern side; for the classics develop the power of sustained and orderly thinking!" Our conclusion is, that the power of sustained and close observation, as required by the chemist and the biologist, is checked by classical studies. The writer seems to detect objections to the study of physiology which strike us as singular. The "honest boy" discussed on p. 275 must be a morbid, and we believe a rare, phenomenon.

The objections urged against zoology, as tending "to become uncleanly," cannot apply to its widest, most interesting, and at the same time most utilitarian department—entomology.

The writers of these "Chapters" merit full recognition as being unfriendly to the modern educational idol, examinationism. Hence we must conscientiously and warmly recommend it to the heads of colleges.

The University of Nebraska. Calendar, 1896-1897.

THE University of Nebraska is in some respects exceptional in its constitution. Unlike the German universities, but like those of Britain, it has what we may venture to call a faculty of music. Unlike all the European seats of learning, it includes painting, sculpture and architecture, and elocution and oratory in its scope. In other words, according to Section 521 of its Rules, it comprises a college of literature, science, and art; secondly, an industrial college, embracing agriculture, practical science, civil engineering, and the mechanical arts; third, a college of law; fourthly, a college of medicine; fifthly, a college of the fine arts.

Section 521 enacts that no person shall—because of age, sex, colour, or nationality—be excluded from the privileges of this institution. This sweeping proviso might, we submit, be amended by fixing a junior limit of age for matriculation. For "colour" we would, had we the right to interfere, substitute "race."

There is here, as it appears to us, too great a tendency to commit different duties to one and the same person. Thus we find a professor of entomology, ornithology, and *taxidermy*. Taxidermy is generally the duty of the curator or "custos." We perceive that there are at the Nebraska University a chemical laboratory, a philosophical laboratory, and a physical laboratory, as well as a botanical and an electrical. It must be at times difficult to define the scopes and duties of the different laboratories.

The "sugar school" confines its attention to practical study of the beet sugars.

A branch of study, concerning which we feel in some doubt, is known as "civics," and, considered in conjunction with political economy, "electives" may mean optional courses of study.

There is also a department for military studies, doubtless for tactics and strategy.

New Process of Sterilisation by Heat under Pressure.—W. Kühn.—The author's experiments show that when the conditions of heating prevent all loss of gas and of aromatic and volatile principles the liquid is unchanged in its argonoleptic properties.—*Compt. Rend.*, cxxiv., No. 9.

CORRESPONDENCE.

THE CHEMICAL SOCIETY ELECTION.

To the Editor of the Chemical News.

SIR,—At the Chemical Society meeting, March 18, 1897, I made the statement that the nomination of Professor W. Ramsay as a candidate for the Presidency of the Chemical Society was made without his knowledge or consent. To the best of my belief, and so far as I have any means of judging, that was an accurate statement. I have seen a letter in the *CHEMICAL NEWS* from Prof. Armstrong in which he "ventures to doubt the accuracy of that statement." I wish to state distinctly that he has no ground whatever for his doubt. When I made my statement I was sure of my facts and I am so still.—I am, &c.,

J. NORMAN COLLIE.

16, Campden Grove, Kensington, W.,
April 1, 1897.

THE NEW SCIENTIFIC CLUB.

To the Editor of the Chemical News.

SIR,—My attention has been called to the fact that my name appears on a circular, signed "Robert Ingram," relating to the proposed formation of a new Scientific Club. I wish to say that the use of my name in this connection is entirely unauthorised. I have no intention of joining any Club promoted by Mr. Robert Ingram.—I am, &c.,

T. E. THORPE.

March 25, 1897.

TEACHING OF CHEMISTRY.

To the Editor of the Chemical News.

SIR,—The importance of this subject is the only apology I offer for re-opening the discussion. Its importance to the rising generation of chemists, manufacturers, &c., is obvious.

Mr. Beebe adduces three reasons why the novice, at the outset of his career, should not devote his time to the preparation of gases, salts, and such like. I will take them in order:—

1. *That they are dangerous.*—Mr. Wigley, who, judging from his letter, must have had a lengthened experience as a teacher of chemistry, says "that there is more danger in acid from a test-tube being boiled over a neighbour's face than in making hydrogen." I agree with him; for, as far as my experience goes, I have never seen any one hurt in the preparation of the latter, whereas several instances have come under my notice in which boiling acid or other liquid has been shot out into a fellow student's face.

2. *Failure in connecting the various experiments.*—I think the boot is on the other foot. There is a great similarity between a good many of the experiments leading from one thing to another. Take as an example KClO_3 , NH_4NO_3 , NH_4NO_2 , $\text{H}_2\text{C}_2\text{O}_4$, and many other substances which, when heated, split up into simpler bodies, whereas in precipitating a substance a more complex compound is often produced.

3. *Does not awaken and hold students' interests like Qualitative Analysis.*—My answer to this is, if the student is guided aright he very soon acquires sufficient powers of observation to find them interesting, and he gets a thorough grounding in the fundamental truths of chemistry whereby he may become a sterling and intelligent chemist. Such has been my experience of students.

Mr. Beebe remarks that lecture experiments are unne-

cessarily complex, and students' ideas confused and hazy. Does not this statement controvert his own objection to the students performing his own experiments?

To commence a beginner's course of chemistry with qualitative analysis is tantamount to teaching a boy who is intended to become a musician the art of turning the handle of a barrel organ. A novice is incapable of intelligently understanding what he does in qualitative analysis, or why he does it. I agree with Mr. Beebe that the first lessons should be as simple as possible. But which is the easier for the young student to understand (I take the simplest instance in each case), $\text{AgNO}_3 + \text{HCl} = \text{AgCl} + \text{HNO}_3$ or $\text{HgO} = \text{Hg} + \text{O}$? I say most decidedly the latter.

I regret to find that Mr. Beebe thinks a man is a "real chemist" when he can work through an arbitrary analytical table and discover what a simple salt is. I have met with men capable of telling most ordinary substances simply by looking at them, and who had no idea of the reaction that occurs when carbon is burnt. These I presume must be more "real chemists" still.—I am, &c.,

C. SORDES ELLIS, A.I.C., F.C.S.,
Late Demonstrator to the Pharm.
Soc. of Great Britain.

The Technical School,
Radcliffe, Lancashire,
March 19, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 10, March 8, 1897.

Researches on the Earths contained in Monazitic Sands.—MM. Schützenberger and Boudouard.—The authors have separated:—1. A cerium of an atomic weight bordering upon 138 or somewhat lower, the solution of which is not precipitated by copper oxide. 2. A cerium of an atomic weight close upon 148, the sulphate of which is precipitable by copper oxide and also by sodium sulphate. 3. A cerium of an atomic weight close upon 157, its sulphate being precipitable by copper oxide, but not by sodium sulphate. The solutions of this sulphate are characterised by the gummy aspect which they take during concentration. These three earths yield yellow ceric salts decomposable by heat into white cerous salts. Hydrogen peroxide with soda precipitates them with an orange-red colour. Ammonium oxalate precipitates all these, and the precipitates are not soluble in a cold excess of the reagent. No difference between the three earths is detected on spectroscopic examination. The portion soluble in neutral ammonium oxalate, and not precipitable by sodium sulphate, does not display the coloured reactions of cerium. It belongs to the thorium group. The experiments indicate that we have not to do with a homogeneous product.

Apparatus employed for Collecting Air at Great Heights in the Ascent of the Aërophile on February 18, 1897. Analysis of the Air collected.—L. Cailletet.—In 100 vols. of air deprived of carbonic acid, and taken at the height of 15,500 metres, there was found—

Oxygen		20.79 vols.
Nitrogen		78.27 "
Argon		0.94 "

The ratio of argon to the total of nitrogen plus argon = 0.01185.

Action of Phosphorus upon Gold.—A. Granger.—The author, after referring to the experiments of Schröter, Hautefeuille, and Perry, states that he has obtained a gold phosphide, Au_3P_4 , of a grey colour and very

brittle. It is easily destroyed if heated in contact with air, and is readily attacked by chlorine and aqua regia.

Determination of Antimony as Peroxide.—H. Baubigny.—This paper will be inserted in full.

Action of Free Bases upon Salts.—Albert Colson.—The decomposition of ammoniacal salts is a phenomenon of heterogeneous dissociation, comparable to the decomposition of lead chloride by sulphuric acid.

A New Derivative of Phenylisindazol obtained by the Action of Salicylic Aldehyd upon Phenylhydrazin.—H. Causse.—The product of the reaction of salicylic aldehyd upon phenylhydrazin has been hitherto regarded as a hydrazone with the melting-point at 142° . The product which we have obtained has a composition agreeing with the formula $\text{C}_{12}\text{H}_{10}\text{N}_2$. When pure and dry it forms stable, colourless needles, which turn green on exposure to light, and melt without decomposition at 142° . Ferric chloride occasions no change of colour, and Fehling's liquid effects no reduction.

Action of Tannin upon some Alkaloids.—Æchsner de Coninck.—The author takes 0.0834 grm. pure dry tannin and 4.610 grms. pure tannin. On mixing the two substances no precipitation is occasioned, though the tannin becomes moist and is gradually dissolved. The liquid is placed above sulphuric acid, when in about three days it deposits a viscid mass, insoluble in cold water. The author introduces, into a very strong aqueous solution of pyridine, a little pure tannin, when there is no precipitation, but the tannin rapidly forms a viscid mass. A solution of tannin serves for the ready distinction of pyridine and piperidine.

Employment of Cryoscopy in the Analysis of Milk.—MM. Bordas and Ganin.—The authors maintain that it is impossible to admit the constant congelation point of milk.

MISCELLANEOUS.

Chemical Society.—Anniversary Meeting.—At the General Meeting held at the Society's Rooms, Wednesday, March 31st, 1897, Professor Dewar, F.R.S., was elected President. The meeting was the fullest on record, between 300 and 400 Fellows being present.

MEETINGS FOR THE WEEK.

- MONDAY, 5th.—Society of Arts, 4.30. (Cantor Lectures). "Alloys," by Prof. W. Chandler Roberts-Austen, F.R.S.
Society of Chemical Industry, 8. Election of Officers and Five Members of Committee. "Chemical Stability of Nitro-compound Explosives," by O. Guttmann, F.I.C.
- TUESDAY, 6th.—Royal Institution, 9. "Animal Electricity," by Prof. A. D. Waller, F.R.S.
Society of Arts, 8. "Recent Travels in Rhodesia and British Bechuanaland," by C. E. Fripp.
- WEDNESDAY, 7th.—Society of Arts, 8. "Dairy Produce and Milk Supply," by M. J. Dunstan, F.R.S.E.
- THURSDAY, 8th.—Royal Institution, 3. "The Relation of Geology to History," by Prof. W. Boyd Dawkins, M.A., F.R.S., F.G.S.
- FRIDAY, 9th.—Royal Institution, 9. "The Limits of Audition," by Lord Rayleigh, F.R.S.
Physical, 5. "A Nickel Stress Telephone," by T. A. Garrett, M.A., and W. Lucas, M.A. "On Alternating Currents in Concentric Conductors," by W. A. Price, M.A. "Effect of Capacity on Stationary Electrical Waves in Wires," by W. B. Morton, M.A.
- SATURDAY, 10th.—Royal Institution, 3. "Electricity and Electrical Vibrations," by Right Hon. Lord Rayleigh, M.A., F.R.S.

TO CORRESPONDENTS.

D. W. Birch.—Write to the author of the paper, care of the Secretary of the Academy of Sciences, 55 Quai des Grands-Augustins, Paris.
H. I. Foster.—Consult "The Practical Methods of Organic Chemistry," by L. Gattermann. Published by Macmillan & Co., 1896.

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1950.

ON THE GASES ENCLOSED IN CRYSTALLINE ROCKS AND MINERALS.*

By W. A. TILDEN, D.Sc., F.R.S.

It has long been known† that many crystallised minerals contain gas enclosed in cavities in which drops of liquid are also frequently visible. The liquid often consists of water, occasionally of hydrocarbons, and not unfrequently of carbon dioxide, the latter being recognisable by the peculiarities of its behaviour under the application of heat. The liquid supposed to be carbon dioxide has been found in some cases to pass from the liquid to the gaseous state, and therefore to disappear, and to return from gas to liquid at temperatures lower by two or three degrees than the critical point of carbon dioxide. This seems to indicate the presence of some incondensable gas, and as H. Davy found nitrogen in the fluid cavities of quartz, it seemed probable that the alteration of the critical-point was due to that gas.

My attention was drawn to this subject by the observation that Peterhead granite, when heated in a vacuum, gives off several times its volume of gas, consisting, to the extent of three-fourths of its volume, of hydrogen (*Roy. Soc. Proc.*, vol. lix., p. 218).

Since this observation, I find that the presence of hydrogen in crystalline rocks has been recognised by other observers, notably by A. W. Wright (*Amer. J. Sci.*, vol. xii., p. 171). In the course of a study of the gases from meteorites, Wright obtained from a certain "trap" rock, the origin and character of which is not stated, at a low red heat, "about three-fourths of its volume of mixed gases, which were found to contain about 13 per cent of carbon dioxide, the residue being chiefly hydrogen. Another specimen of trap containing small nodules of anorthite was examined at the request of Mr. G. W. Hames, who had observed gas cavities in a thin section of the mineral prepared for microscopic examination. This gave off somewhat more than its own volume of gas, which was found to contain some 24 per cent of carbon dioxide."

Professor Dewar and Mr. Ansdell have also examined one or two rocks in the course of their researches on meteorites (*Roy. Inst. Proc.*, 1886). They found that both gneiss and felspar, containing graphite, yield gas, which, upon analysis, was found to have the composition stated below:—

	Occluded gas in volumes of the rock.	CO ₂ .	CO.	H ₂ .	CH ₄ .	N ₂ .
Gneiss ..	5'32	82'38	2'38	13'61	0'47	1'20
Felspar ..	1'27	94'72	0'81	2'21	0'61	1'40

Dewar and Ansdell remark that "the small quantity of marsh gas, no doubt, comes from the disseminated graphite, but the presence of the hydrogen is more difficult to explain, and requires further investigation."

I have lately been following up this question, and have obtained results which present some points of considerable interest. For materials I have been indebted chiefly to my colleague, Professor Judd, who has also supplied in-

formation as to the probable geological age of the specimens of rocks and minerals tested. All that I have examined yield permanent gas when heated in a vacuum. This gas varies in amount from a volume about equal to that of the rock or mineral to about eighteen times that volume. It usually consists of hydrogen in much larger proportion than that found by the observers just quoted, together with carbon dioxide and smaller quantities of carbon monoxide and hydrocarbons. Every specimen has been examined by the spectroscope for helium, but in no case could D₃ be recognised, or any other line which would lead to a suspicion of the presence of this substance. The gas is very frequently, but not always, accompanied by water in notable quantities.

The gas is apparently wholly enclosed in cavities which are visible in thin sections of the rock when viewed under the microscope, but as they are extremely minute, very little gas is lost when the rock is reduced to coarse powder, and as a result of experiment in one or two cases, I find that practically the same amount of gas is evolved on heating the rock whether it is used in small lumps or in powder. In the first series of experiments undertaken with the object of a rapid survey of the materials, the gases were not completely analysed. They were collected, measured, the carbon dioxide removed by potash, and the residue examined by the spectroscope. When ignited in the air it always burned with a pale flame resembling that of hydrogen.

The accompanying table gives the results of these experiments.

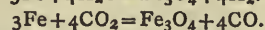
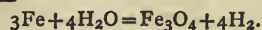
A selection of these was then subjected to more careful and exact analysis. For this purpose fresh masses of the rock were taken, and the gas extracted in the usual way. The following are the results:—

	CO ₂ .	CO.	CH ₄ .	N ₂ .	H ₂ .
Granite from Skye ..	23'60	6'45	3'02	5'13	61'68
Gabbro from Lizard ..	5'50	2'16	2'03	1'90	88'42
Pyroxene gneiss, Ceylon ..	77'72	8'06	0'56	1'16	12'49
Gneiss from Seringapatam ..	31'62	5'36	0'51	0'56	61'93
Basalt from Antrim ..	32'08	20'08	10'00	1'61	36'15

To account for the large proportion of hydrogen and carbonic oxide in these gases, it is only necessary to suppose that the rock enclosing them was crystallised in an atmosphere rich in carbon dioxide and steam which had been, or were at the same time, in contact with some easily oxidisable substance, at a moderately high temperature. Of the substances capable of so acting, carbon, a metal, or a protoxide of a metal, present themselves as the most probable.

The reduction of carbon dioxide or of water vapour by carbon gives rise to the formation of carbon monoxide, and if carbon had been the agent the proportion of this gas in the mixture must have been greater than is found to be the case. It is, of course, well known that carbon dioxide and water vapour are both dissociated at moderately high temperatures, but the greater part of the liberated oxygen re-combines at lower temperatures, though a small portion may remain free in the presence of a large quantity of an indifferent gas or vapour. No free oxygen has been found in any of the gases analysed.

Direct experiments made with ferrous oxide (obtained by gently heating pure chalybite) and with magnetic oxide of iron, show that while the former, at a red-heat, decomposes both steam and carbon dioxide quite freely, liberating hydrogen and carbon monoxide, and becoming itself oxidised into magnetic oxide; the latter has no action at all upon either steam or carbon dioxide. Magnetic oxide of iron is the final product of the action of steam or of carbon dioxide at a high temperature upon metallic iron:—



Now, metallic iron has been detected in basalts and some other rocks by Andrews (*Brit. Assoc. Rep.*, 1852,

* A Paper read before the Royal Society, March, 1897.

† The chief literature of this subject is contained in the following papers:—Brewster, *R. S. Edin. Trans.*, 1824, vol. x., p. 1; *Edin. J. Science*, vol. vi., p. 115; Simmler, *Pogg. Ann.*, vol. cv., p. 460; Sorby and Butler, *Roy. Soc. Proc.*, vol. xvii., p. 291; Vogelsang and Geissler, *Pogg. Ann.*, vol. cxxxvii., pp. 56 and 257; Hartley, *C. S. Trans.*, 1876, vol. i., p. 137, and vol. ii., p. 237.

Name of rock or mineral.	Locality.	Character.	Volume of gas per volume of rock.	Composition in 100 volumes.	
				CO ₂ .	H ₂ , &c.
Rocks of Tertiary Age.					
Granite 1	Skye	Plutonic, acid ..	1.6	11.5	88.5
" 2	"	"	2.8	31.0	69.0
"	Salen, Mull.	"	1.3	34.7	65.3
Gabbro.. .. .	L. Coruisk, Skye	" basic..	3.5	21.6	78.4
Basalt	Antrim.. .. .	"	8.0	32.0	68.0
Rocks of Palæozoic Age.					
Quartzite	Durness (Sutherland) . ..	Aqueous, altered	2.2	14.3	85.7
Gabbro.. .. .	Lizard	Plutonic, basic..	6.4	trace	100.0
Granite	Peterhead	" acid ..	2.6	24.8	75.2
"	Cornwall	"	4.3	8.8	91.2
Rocks of Unknown Age (mostly Archæan).					
Granite	Near Dublin	Plutonic, acid ..	5.0	9.4	90.6
"	Ardshiel	"	6.9	79.5	20.5
Greisen	Altenburg (Saxony)	" altered	1.8	13.6	86.4
Granulite	Central India	"	2.6	48.7	51.3
Quartz schist	Cas. Wellan (Co. Down)..	Metamorphic ..	2.8	23.0	77.0
Fuchsite schist	Baroda.. .. .	"	4.2	20.8	79.2
Corundum rock	Pipra, S. Rewah, India	"	3.5	26.0	74.0
Pyroxene gneiss.. .. .	Dombra (Ceylon)	"	7.3	84.4	15.6
Gneiss with corundum	Seringapatam	"	17.8	18.0	82.0
" " garnets and graphite	Doloswella (Ceylon).. ..	"	4.5	11.0	89.0
"	Himalayas (Nanga Parbut)	"	7.2	11.5	88.5
Recent Lava.					
	Vesuvius, 1760	—	0.65	72.0	28.0
Minerals.					
Graphite	Ceylon	—	7.5	48.0	52.0
Quartz matrix of same	—	—	1.2	44.5	55.5
Beryl	Irish	—	6.7	6.0	94.0
Tinstone	Straits Settlements	—	1.3	45.4	54.6

Sections, p. 34), and by some other observers (e.g., G. W. Hawes, *Amer. J. Sci.*, 1877, Ser. 3, vol. xiii., p. 33), and I have verified this observation in the case of the gabbro of Loch Coruisk. But it must be remembered that both the reactions indicated in the equations just given are reversible, and therefore the presence of metallic iron along with the magnetic oxide in such rocks cannot be taken by itself as final proof that the oxide and the associated gases, hydrogen and carbonic oxide, are the products of the action of steam and carbon dioxide upon metallic iron. The presence of marsh gas in these rocks and the production of large quantities of hydrocarbonous gases, as well as liquid petroleum, in many parts of the earth's surface, tend to support the view, which is apparently gaining ground, that in the interior of the earth's crust there are large masses, not only of metal but of compounds of metals, such as iron and manganese, with carbon. Assuming the existence of such material, it is easy to conceive how, by the action of water at an elevated temperature, it may give rise to metallic oxides and mixtures of hydrogen with paraffinoid and other hydrocarbons. This view was put forward some years ago by Mendeleeff ("Principles of Chemistry," Translation by Kamensky and Greenaway, vol. i., 364—365), and it has lately received further support from the results of the study of metallic carbides, which we owe especially to Moissan (*Roy. Soc. Proc.*, vol. ix., 1896, pp. 156—160).

Determination of Phosphate in Thomas Slags.—According to Dr. O. Böttcher (*Chemiker Zeitung*), the citrate method cannot be universally accepted for the determination in ground Thomas slags of phosphate soluble in citric acid until further comparative analyses have been carried out.

DETERMINATION OF ATMOSPHERIC OZONE ON MONT BLANC.

By MAURICE DE THIERRY.

THANKS to the kind support of M. Janssen I have been able to commence, in 1894, a series of researches on Mont Blanc, and continue them in 1895 and 1896. The bad weather, which has not ceased to prevail on these heights during last summer, has hindered me from completely executing the programme which I had drawn up; still the interest which the first part of my researches seemed to present encourages me to present them to the Academy.

In September, 1894, I was struck with the rapidity with which slips of amidised ozonoscopic paper (Schœnbein's paper), and red litmus paper saturated with potassium iodide (Houzeau's paper), exposed to the air on the platform of the Observatory on the summit of Mont Blanc, at the altitude of 4812 metres, took respectively deep violet and blue colourations. Paper steeped in thalious oxide was equally and rapidly blackened with formation of thallic oxide (Bœttgen reaction). It was the same with plates of silver which I had prepared by reducing a solution of silver nitrate with lactose.

These first observations were already interesting when—on Tuesday, August 13th, at noon—I, along with my two guides, was attacked by a tempest of snow (altitude of 42,000 metres), accompanied by numerous peals of thunder, and the fall of hailstones perfectly spherical and of the size of large peas.

A violent N.W. wind was blowing; Naudin's air hygrometer marked 115°, and the thermometer, which before the storm was at 0°, fell rapidly to -15. A number of hailstones, collected on a sheet of iodo-amidised

ozonoscopic paper, made immediately circular violet spots, larger than the diameter of the hailstones, the centre of the spots occupied by the hailstones being paler than the circumference. The violence of the storm did not allow me to examine closely if these spots were formed by an atmosphere of ozone surrounding the hailstone when it fell, or by hydrogen peroxide (which might give the same action) derived from the melting of the hail. I must add that the snows (recent or old) taken on adjoining heights have never given the characteristic reactions of hydrogen peroxide. The water from melted snow has always given the characteristic reaction of ammonia with Nessler's test.

The author resolved to determine quantitatively the atmospheric ozone on Mont Blanc. He finds that at Chamounix (1050 metres) the atmospheric ozone is 3·5 m.grms., and on the Grand Mulets (3020 metres) 9·4 m.grms. per 1000 cubic metres of air,—that is to say, nearly four times greater than at Paris. The quantity of ozone therefore increases with the altitude.

The author is engaged with the analysis of air which he has brought with him from the Grand Mulets. He is also studying the possible presence of nitrous acid in certain strata of air.—*Comptes Rendus*, cxxiv., No. 9.

EXPERIMENTS ON THE ACTION OF PERMANGANATE OF POTASH AND ACETIC ACID ON THE BACTERIA IN RAW THAMES WATER.

By HENRY CROOKES, A.R.S.M., M.I.E.E.

A FEW months ago I carried out a series of experiments on the action of certain germicides on the bacteria in raw Thames water, my primary object being to determine the influence of time, and quantity of material employed.—After a number of tentative and preliminary experiments, to get an idea of the most suitable quantities and strengths, I at length decided to work with a constant volume of 250 c.c. of raw water, adding to it a standard solution of KMnO_4 of 20 grains per gallon. The method of procedure was as follows:—

In the first series of experiments 10 c.c. of the standard KMnO_4 were added to three different bottles, each containing 250 c.c. of unfiltered water; these were marked B, C, D, while A was the raw water untreated.

B was allowed to stand for 15 minutes, when 0·5 c.c. was mixed with 10 c.c. of nutrient gelatin peptone, poured into a sterile Petri dish, and when cool placed in the incubator, and kept at 20° to 20·5° for 48 hours.

C was allowed to stand for 30 minutes, and D for 60 minutes, both these (as well as A) being then treated in exactly the same manner as B. After 48 hours the colonies were counted under the microscope, and the following results obtained:—

SERIES I.	Colonies per c.c.
A. Unfiltered water untreated	3876
B. 250 c.c. of same water + 10 c.c. standard KMnO_4 , after 15 minutes	332
C. 250 c.c. of same water + 10 c.c. standard KMnO_4 , after 30 minutes	240
D. 250 c.c. of same water + 10 c.c. standard KMnO_4 , after 60 minutes	36

These figures show a very rapid diminution in the number of bacteria, even with so weak a solution of KMnO_4 as 20 grains per gallon.

My next experiments were to determine the germicidal power of varying quantities of KMnO_4 (always the same standard strength) in a constant time. Four bottles were taken, as before, A being untreated; to B were added 5 c.c., to C 10 c.c., and to D 20 c.c. of KMnO_4 ; these

were all allowed to stand for 15 minutes, then treated as before, and incubated for 48 hours. At the end of that time the following number of colonies per c.c. were found:—

SERIES II.	Colonies per c.c.
A. Unfiltered water untreated	3850
B. 250 c.c. of same water + 5 c.c. standard KMnO_4 , after 15 minutes	260
C. 250 c.c. of same water + 10 c.c. standard KMnO_4 , after 15 minutes	190
D. 250 c.c. of same water + 20 c.c. standard KMnO_4 , after 15 minutes	40

From these figures it would appear that there is very little difference in the action of a small quantity of KMnO_4 for a long time, and a much larger quantity for a shorter time.

Another set of experiments conducted in exactly the same manner, save that the standard KMnO_4 was made slightly acid with acetic acid, gave the following results:—

SERIES III.	Colonies per c.c.
A. Unfiltered water, untreated	2040
B. 250 c.c. of same water + 10 c.c. standard KMnO_4 , after 15 minutes	116
C. 250 c.c. of same water + 10 c.c. standard KMnO_4 , after 30 minutes	90
D. 250 c.c. of same water + 10 c.c. standard KMnO_4 , after 60 minutes	55

SERIES IV.	Colonies per c.c.
A. Unfiltered water, untreated	2400
B. 250 c.c. of same water + 5 c.c. standard KMnO_4 , after 15 minutes	589
C. 250 c.c. of same water + 10 c.c. standard KMnO_4 , after 15 minutes	141
D. 250 c.c. of same water + 20 c.c. standard KMnO_4 , after 15 minutes	90

Finally, another set was done, in all respects similar, but that the standard KMnO_4 was made alkaline with caustic soda, the results being as follows:—

SERIES V.	Colonies per c.c.
A. Unfiltered water untreated	3080
B. 250 c.c. of same water + 10 c.c. of standard KMnO_4 , after 15 minutes	268
C. 250 c.c. of same water + 10 c.c. of standard KMnO_4 , after 30 minutes	120
D. 250 c.c. of same water + 10 c.c. of standard KMnO_4 , after 60 minutes	87

SERIES VI.	Colonies per c.c.
A. Unfiltered water untreated	12400
B. 250 c.c. of same water + 5 c.c. of standard KMnO_4 , after 15 minutes	3720
C. 250 c.c. of same water + 10 c.c. of standard KMnO_4 , after 15 minutes	2170
D. 250 c.c. of same water + 20 c.c. of standard KMnO_4 , after 15 minutes	434

These six series of experiments, when calculated as percentages of microbes present at each stage, appear as follows, and can be easily compared:—

SERIES	Neutral.		Acid.		Alkaline.	
	I.	II.	III.	IV.	V.	VI.
A.	100·0	100·0	100·0	100·0	100·0	100·0
B.	8·5	6·7	5·7	24·5	8·7	30·0
C.	6·1	4·9	4·4	6·0	3·9	17·5
D.	0·9	1·0	2·7	3·7	2·8	3·5

We see at once that the strongest and most rapid action takes place when the permanganate solution is neutral, the most noticeable difference being the remarkable loss of power of 5 c.c. of both acid and alkaline permanganate (Series IV. and VI.) when acting for fifteen minutes (B).

I next turned my attention to the action of acetic acid alone on unfiltered water; the methods of procedure and incubation were the same as in the previous experiments, the first being with a varying quantity of a 1 per cent solution standing for 15 minutes. After 48 hours in the incubator the following results were obtained:—

SERIES VII.	Colonies per c.c.
A. Unfiltered water untreated	5890
B. 250 c.c. of same water + 1 c.c. of 1 per cent acid, after 15 minutes	3684
C. 250 c.c. of same water + 5 c.c. of 1 per cent acid, after 15 minutes	3262
D. 250 c.c. of same water + 10 c.c. of 1 per cent acid, after 15 minutes	2846

These figures show a slight and regular diminution in the number of bacteria; but the acid was evidently too weak, so a stronger solution—viz., 10 per cent—was prepared and the experiments repeated with the following results:—

SERIES VIII.	Colonies per c.c.
A. Unfiltered water untreated	824
B. 250 c.c. of same water + 1 c.c. of 10 per cent acid, after 15 minutes	600
C. 250 c.c. of same water + 5 c.c. of 10 per cent acid, after 15 minutes	456
D. 250 c.c. of same water + 10 c.c. of 10 per cent acid, after 15 minutes	238

These showed a stronger action, but nothing striking, so a further lot was done with a 50 per cent solution of acetic acid, with the following remarkable results:—

SERIES IX.	Colonies per c.c.
A. Unfiltered water untreated	1976
B. 250 c.c. of same water + 1 c.c. of 50 per cent acid, after 15 minutes	750
C. 250 c.c. of same water + 5 c.c. of 50 per cent acid, after 15 minutes	30
D. 250 c.c. of same water + 10 c.c. of 50 per cent acid, after 15 minutes	0

A 50 per cent solution of acetic acid proving unnecessarily strong, two more trials were made with the 10 per cent solution, keeping the quantities constant, viz., 5 c.c. and 10 c.c., and varying the time 10, 30, and 60 minutes.

SERIES X.	Colonies per c.c.
A. Unfiltered water, untreated	9792
B. 250 c.c. of same water + 5 c.c. of 10 per cent acid, after 10 minutes	4096
C. 250 c.c. of same water + 5 c.c. of 10 per cent acid, after 30 minutes	2176
D. 250 c.c. of same water + 5 c.c. of 10 per cent acid, after 60 minutes	1280

SERIES XI.	Colonies per c.c.
A. Unfiltered water, untreated	2090
B. 250 c.c. of same water + 10 c.c. of 10 per cent acid, after 10 minutes	508
C. 250 c.c. of same water + 10 c.c. of 10 per cent acid, after 30 minutes	380
D. 250 c.c. of same water + 10 c.c. of 10 per cent acid, after 60 minutes	280

By converting the results of Series IX., X., and XI. into percentages as before, we get the following table:—

	Series IX.	Series X.	Series XI.
A.	100.0	100.0	100.0
B.	37.9	41.8	24.3
C.	1.5	22.2	18.1
D.	0.0	13.0	13.4

From these figures we see that time is a stronger factor in the decrease in the number of microbes than an in-

crease in the quantity of the acid used. Thus, in Series X. and XI., while after ten minutes standing (B) the difference is very marked, after sixty minutes (D) there is practically nothing to choose between them.

Further experiments are being carried on, and I hope to be able to publish the results in due course.

A REVISION OF THE ATOMIC WEIGHT OF MAGNESIUM.*

By THEODORE WILLIAM RICHARDS
and
HARRY GEORGE PARKER.

(Continued from p. 159).

Purification of Silver.

No very great labour was expended upon the purification of the first quantity of silver, as the chlorine in magnesian chloride was to be precipitated with an undetermined excess of silver nitrate. Residues were therefore worked up by dissolving silver (obtained by reduction with zinc) in nitric acid, precipitating the metal as chloride, and converting the chloride into metallic silver by means of invert sugar. The reduced silver, after having been fused into buttons, was thoroughly washed and dissolved in nitric acid. The solution of argentic nitrate thus obtained was diluted very much with water, allowed to stand, and filtered just previous to using.

With the second sample, on the other hand, much greater care was taken, as it was designed in this case to ascertain the direct ratio between silver and magnesian chloride. The material came partly from some refined silver, purchased in the market, and partly from some pure silver residues remaining from previous work. The silver was precipitated from a solution of the nitrate with pure hydrochloric acid, and reduced by means of invert sugar and pure sodic hydrate, the sodic hydrate having been previously freed from heavy metals by electrolysis. Both the chloride and reduced silver were very thoroughly washed, the silver was dissolved in pure nitric acid, and the process was repeated. After this cycle of operations had been performed four or five times, the reduced silver was fused on a cupel of sugar charcoal before the blow-pipe. The resulting button was scrubbed with sand, and made the anode of a weak galvanic circuit in a solution of argentic nitrate prepared from the same silver. The cathode was a piece of pure silver wire, upon which the whole of the silver was deposited in a crystalline mass. The silver crystals were then removed from the solution and fused in a vacuum upon a boat of pure lime (*Proc. Amer. Acad. Arts Sci.*, xxx., 379; xxxi., 173), which was contained in a porcelain tube. Such a boat may be made by lining a porcelain boat with a mixture of three parts of pure lime and one part of pure anhydrous calcic nitrate, and igniting the mixture. The porcelain boat is thus covered with a firm coherent layer of pure calcic oxide. In order to prevent the possibility of a trace of organic matter distilling off from the rubber stoppers usually used to close such a tube, a set of hollow brass stoppers were made, through which a current of cold water circulated. This latter device is due to a suggestion of Professor Hempel. The construction of this piece of apparatus is evident from the diagram (Fig. 2).

Of course, the button after fusion showed no trace of spiriting from contained oxygen. It was scrubbed with distilled water and clean sand, and divided into small pieces by means of a clean steel chisel. The fragments were alternately boiled in strong hydrochloric acid and digested in ammonia water, this process being repeated ten or fifteen times. The silver was finally washed with

* Contributions from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, vol. xxxii., No. 2.

distilled water and afterwards kept in a desiccator, which was opened only when necessary to weigh out silver for a determination.

A portion of the second sample was treated in the same way, except that in the end it was fused on sugar charcoal before the blowpipe and cooled in the reducing flame. Particular pains were taken to prevent the absorption of oxygen, and the button did not show the slightest trace of having contained this gas. From this portion wire was prepared of various thicknesses, by means of a draw plate; and the weights of given lengths of these wires were determined, so that small weights could be made with considerable accuracy. Of course, the wire was treated in the same fashion as the rest of the silver, in order to remove any iron which might be present on the surface.

The third and fourth samples of silver were prepared in the same manner as the second, the starting-point being the pure residues left from the analyses made with previous samples. No qualitative nor quantitative difference could be observed between any of these preparations of silver. Fused upon sugar charcoal, they melted to a clear globule free from any film,—a fact which in itself, according to Stas, is an excellent test of the purity of silver,—and all gave practically the same results in later determinations.

All water used was re-distilled with potassic perman-

xxx., 369; xxxi., 158). We are indebted to the Cyrus M. Warren Fund of Harvard University for some of the platinum ware used in the following work.

The atomic weights used in this investigation were as follows:—

O	16.000	Ag	107.930
Cl	35.456		

Method of Work.

The method of operating may be inferred from the description of the apparatus. The platinum boat, after having been weighed within its weighing bottle, was filled with the double chloride of ammonium and magnesium and placed in position in the ignition-tube, resting upon a sort of carriage of platinum foil. The weighing bottle was placed with its stopper in appropriate position in the "bottling tube," as previously described. A current of dry hydrochloric acid gas was then passed through the apparatus and the ignition tube was heated by a suitable arrangement of burners. At first the residual moisture was driven off by the heat and carried away by the stream of gas. When as much water as possible was expelled in this manner, the heat was slowly increased, so that the ammonic chloride commences to vaporise. It was found that the sublimation commenced before the salt was freed from the last traces of moisture, but an effort was always

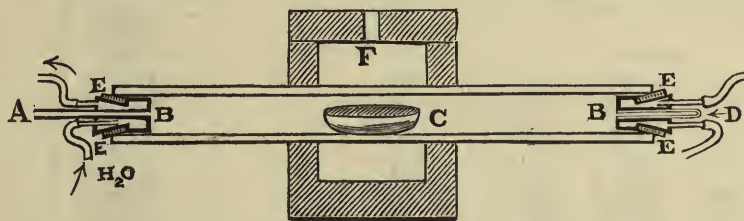


FIG. 2.—APPARATUS FOR FUSING SILVER, VERTICAL SECTION.

A is connected with a Sprengel pump. B B, hollow brass stoppers in porcelain tube. C, boat of lime containing silver. D, "window" for observation. E E E, rubber packing of stopper. F, Fletcher furnace.

ganate, some of it being condensed in a platinum condenser, and some of it by means of a tube of pure block tin, which was carefully tested in order to prove the absence of an impurity of lead. Considerable quantities upon evaporation in platinum left a scarcely appreciable residue, there being apparently no difference between the water condensed in tin and that in platinum (see *Proc. Amer. Acad. Arts Sci.*, xxvi., 249; xxx., 380). The water was prepared as short a time as possible before being used, and was carefully kept in a suitable bottle fitted with a syphon, air being admitted to the bottle through a filter of cotton-wool. It was carefully tested for chlorine by means of the nephelometer from time to time.

The sulphuric acid used for the preliminary drying of the gases was the usual "chemically pure" acid of the laboratory, of a specific gravity of about 1.83. For the final drying this acid was boiled down in platinum.

Weighing.

The balance used was a long-armed Becker, sensitive to about one-thirtieth of a milligram, with the largest load that it was required to carry during the investigation, while the weights were a good set of gold plated ones, which were kept in the balance case under a glass cover. These weights were very carefully compared with one another, and all weighings were, of course, reduced to the vacuum standard. The specific gravity of magnesian chloride used for this computation was the value 2.177 determined by Playfair and Joule. Weighing was done by substitution, the object to be weighed being placed on the right-hand pan and balanced by tare weights on the left. In general, the precautions used in the recent work done in this laboratory upon copper, barium, strontium, and zinc were adhered to with great care (Richards, *Proc. Amer. Acad. Arts Sci.*, xxvi., 240; xxviii., 1; xxix., 55;

made, by the very gradual increase of heat, to make this proportion of water as small as possible; and it is probable that the salt was practically anhydrous sometime before the last of the ammonic chloride was sublimed. When no further evolution of ammonic chloride could be observed, the heat was increased until the tube and boat were heated to redness, and the magnesian chloride had fused into a clear, colourless limpid liquid. It requires a very excellent piece of combustion tubing to stand the heat necessary to fuse magnesian chloride, and a number of tubes were spoiled during the course of the work. In the first series of determinations the boat was allowed to cool while the current of hydrochloric acid gas was still passing. The tubes containing the boat and weighing bottle were then thoroughly washed out with a current of air dried in an apparatus similar to that used for drying the hydrochloric acid gas, as previously described. After it was certain that all of the acid gas had been displaced, and while the current of air was passing rapidly to prevent any diffusion of moist air back into the apparatus, the bulbs were removed from the farther end of the ignition-tube, and the boat was pushed into the bottle in the manner already described. The boat itself remained constant in weight during these operations, showing that the magnesian chloride had not acted upon it.

After weighing, the boat and its contents were placed in a large glass-stoppered Erlenmeyer flask, and the magnesian chloride was dissolved in pure water. The chlorine was precipitated with a dilute solution of argentic nitrate (this solution contained never more than 1 per cent of silver); and after a thorough shaking the whole was allowed to stand in the dark over night. The argentic chloride was washed by decantation a number of times, with vigorous shaking, and was finally collected upon a Gooch crucible in the usual manner. The precipitate

was dried from five to ten hours in an oven, carefully protected from dust and dirt, and weighed. After weighing, the cake of precipitate, together with some adherent asbestos, was removed to a tared porcelain crucible and heated until it began to fuse. The crucible was again weighed, and the loss of weight, if any, noted, and subtracted from the weight of the Gooch crucible and contents. The filtrate, containing a little dissolved argentic chloride, was evaporated down to small bulk and filtered through a very small filter; and the weight of the precipitate was added to the weight of the first portion. In some cases the small amount of argentic chloride present was determined with the nephelometer (see *Proc. Amer. Acad. Arts Sci.*, xxx., 385).

The wash water from the precipitate collected on the Gooch crucible was also run through a small filter to make sure that no asbestos had been carried away from the crucible in the process of washing; and this correction, when appreciable, was applied in the appropriate place.

The washing and filtration were both performed in dim orange light, which had been suitably tested as to its non-actinic properties. Even after fusing the argentic chloride was almost colourless, showing that only unessential traces had been decomposed by the light.

The result of the first series of five experiments is given below. These determinations were consecutive, except that one determination met with an accident and was not completed.

SERIES I.

Nö.	Sample of MgCl ₂ used.	Sample of Ag used.	Weight of MgCl ₂ .	Weight of AgCl.	Ratio. MgCl ₂ : 2AgCl = 100 : n.	Atomic weight of Mg.
1	I	I	1'33550	4'01952	300'975	24'368
2	I	I	1'51601	4'56369	301'033	24'350
3	I	I	1'32413	3'98528	300'974	24'369
4	I	I	1'40664	4'23297	300'928	24'384
5	I	I	1'25487	3'77670	300'963	24'373

Average.. .. 24'369

A careful consideration of the possible constant errors involved in the foregoing results lead to the belief that the figures found are too high rather than too low, as the presence either of a small amount of water or of oxy-chloride in the magnesian chloride would tend in this direction.

Second Series of Determinations.

In order to drive all the subliming ammoniac chloride to the further end of the combustion-tube during the ignition, it had been found necessary that the current of gas should be very considerable; and hence it was desirable to construct a piece of apparatus which should deliver the various gases rapidly, but nevertheless as dry as it is possible to obtain them. It was also desirable to work with larger quantities of materials than could be handled in the former apparatus. For these reasons another piece of apparatus was constructed to dry the hydrochloric acid gas; this apparatus contained several flasks of sulphuric acid, three very efficient towers containing the same acid, which was constantly renewed, and a long tube containing re-sublimed phosphoric pentoxide. One of the towers is shown (Fig. 3). The whole apparatus was fused or ground together, thus wholly avoiding rubber or cork connections.

In the following determinations the boat was allowed to cool in an atmosphere of dry nitrogen, as a further precaution against a possible partial decomposition of the sensitive magnesian chloride. As soon as the salt had been fused, a current of dry nitrogen was passed into the combustion-tube and the hydrochloric acid generator was disconnected. The nitrogen was prepared by passing mixed air and ammonia over rolls of copper gauze heated to redness, the excess of ammonia being removed by passing the gases through wash bottles containing dilute sulphuric acid and the nitrogen was dried in a set of

towers similar to those used for drying the current of air. When the tube was cool, the current of dry air was turned on, and the tube and its contents washed out as in previous experiments.

As there were no especial objections against the use of rubber connections and stoppers in the part of the apparatus used for drying the air, several large towers were employed, each filled with crushed pumice-stone and saturated with sulphuric acid previous to using. Both air and nitrogen were finally dried by re-sublimed phosphoric pentoxide. The bottling and combustion-tubes were of the same construction as in the former apparatus, except that they were larger.

In the second series the method of igniting the double salt to obtain the magnesian chloride was the same as in the first; but the method of estimating the amount of chlorine present was different. From the approximate

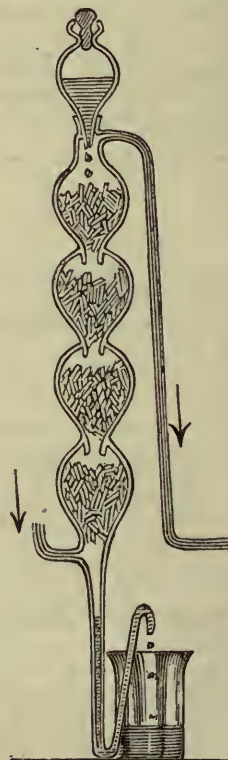


FIG. 3.—ONE OF THE TOWERS USED FOR DRYING HYDROCHLORIC ACID (70 c.m. high).

atomic weight of magnesium already found, a calculation was made as to the amount of silver necessary exactly to precipitate the chlorine present in the sample of magnesian chloride taken. This amount of silver was weighed out as nearly as possible, dissolved in nitric acid in an Erlenmeyer flask, provided with a set of bulbs to catch the spray from the evolution of gas, and added to the solution of magnesian chloride contained in a large flask. The flask was thoroughly agitated in the dark, and allowed to stand over night. Fifty c.c. were then withdrawn by means of a pipette, and tested by means of a nephelometer, or apparatus for determining the amount of precipitate from the intensity of the opalescence produced by it. This piece of apparatus was constructed for the purpose, and consisted of two rectangular glass cells, with a mirror enclosed in a dark case, so arranged that the column of liquid contained in the lower part of the cells could be viewed horizontally without disturbance from

surface reflections. A dark screen was placed at the further end of the cells, and the whole so arranged that light could come to the eye only by reflection from solid particles which might be suspended in the column of liquid inspected. If the liquid was perfectly clear, the field of vision remained black, but an extremely small amount of precipitate produced a very marked change, and the intensity of opalescence was approximately proportional to the amount of precipitate. It was found perfectly easy and certain, by this method, to distinguish the difference between 0.002 and 0.003 of a milligram of argentic chloride or between 0.004 and 0.005 of a milligram, and larger amounts in proportion. This instrument gave such satisfaction in this research that the method will be worked out for various other reactions, and published later.

The method of using this apparatus was as follows:—25 c.c. of the clear supernatant liquid from the flask containing the well-shaken argentic chloride and magnesian nitrate were placed in each cell, 5 c.c. of a very dilute solution of argentic nitrate being added to one, and 5 c.c. of a correspondingly dilute solution of ammoniac chloride to the other. The silver solution contained 1 m.grm. of silver to the c.c. An unequal depth of cloudiness indicated an excess of either silver or chlorine in the original solution, and accordingly the amount necessary for neutralisation was run into the large flask containing precipitate and solution from a burette. The solution was again allowed to stand in the dark with occasional shaking, and after the precipitate had entirely subsided was again tested as before, and this cycle of operations was repeated until the opalescences matched one another.* It will be observed that, if water is added to the cell giving the more dense opalescence until the effect becomes equal on both sides, the amount of dilution will give a means of ascertaining the amount of precipitate in each cell. The appropriate corrections were then applied to the amount of silver taken. Due allowance was made for the slightly diminishing volume of the solution in the flask. The addition of 1-10th of a m.grm. of silver to a litre of solution produced a distinct change in the depth of colour observed. After the matching was completed, repeated trials were made with fresh portions of the solution to confirm the result; and as the depth of opalescence as seen in the nephelometer was perfectly flat, without disturbing effects, the end point could be determined with great precision.

Several results obtained in this manner are given in Series II.

SERIES II.

No. exp.	Sample of MgCl_2 used.	Sample of Ag used.	Weight of MgCl_2 .	Weight of Ag.	Ratio. $\text{MgCl}_2 : 2\text{Ag} = 100 : x$.	Atomic weight of Mg.
6	1	1	2.78284	6.30284	226.490	24.395
	1	1	2.29360	5.19560	226.526	24.379
8	1	2	2.36579	5.35989	226.558	24.366

Average.. .. 24.380

These results, however, do not merit great confidence for the apparatus, which had become somewhat complicated, did not work smoothly at first, on account of some minor imperfections which were remedied later. Besides this, careful consideration led to the suspicion that the towers used for drying the air and nitrogen were not efficient enough to remove the last traces of water. Of necessity the towers had to be charged with sulphuric acid an hour or two before their final use, and during that time a large part of the acid drained out of the pumice stone. This surmise was fully confirmed by later experiments; and since this was the case, the second series must be rejected in the final estimate of the atomic weight.

(To be continued).

THE ATOMIC WEIGHT OF JAPANESE TELLURIUM.

By MASUMI CHIKASHIGE, Rigakushi, College of Science, Imperial University.

THE atomic weight of tellurium has been determined by Berzelius (1833), von Hauer (1857), Wills (1879), Brauner (1883, 1889), and Staudenmaier (1895). Berzelius gave it as 128.3 ($\text{O} = 16$). Staudenmaier has only reduced it to 127.6. Brauner had also obtained this number, that is, 127.64, by determining the quantity of bromine in the tetrabromide; but in other ways, which he could not admit to be inaccurate, he obtained widely varying numbers for the atomic weight. To explain these variations, he assumed that what passes for the element tellurium is a mixture or compound. The number 125, which since 1884 has been generally accepted as the atomic weight of tellurium, was suggested by Mendeleeff, but was adopted on the grounds of Brauner's determinations (partly by faulty methods, as he has since ascertained) published in 1883 in Russia. A paper by him, on the atomic weight of tellurium, which appeared last year in the *Journal of the (London) Chemical Society*, supplies no new data. It throws no light upon the causes of the varying results he had previously obtained by different methods, but apparently contains the admission from him at last that, so far as can be determined by known methods, the atomic weight of tellurium is 127.64 (127.7 in vacuo).

The object of the research described in the present communication has been, not to add one more to the above-mentioned determinations of the atomic weight of tellurium, by some modification of a method already employed or by some new method, but to apply Brauner's tetrabromide method to tellurium of utterly different origin from that of what he worked upon. European and American tellurium occurs in association with heavy metals, and might, therefore, when separated from those which are known, still retain unknown elements, in accordance with Brauner's conception. But in Japan tellurium is found in native sulphur, as was discovered by Divers, Shimose, and Shimidzu, in 1883 (*CHEM. NEWS; J. Chem. Soc.*). There occurs, in fact, in this country, a massive, crystalline, red sulphur, a variety of the *selen-sulphur* (Stromeyer) found in the Lipari Isles, in Naples (Phipson), and in the Hawaiian Islands (Dana). It is semi-transparent, and indistinguishable in appearance from native sulphur, except by its beautiful orange colour, and occurs interspersed with simple sulphur in the same blocks. I take from the *CHEMICAL NEWS* the composition of a sample analysed by Divers and Shimidzu:—Tellurium, 0.17; selenium, 0.06; arsenic, 0.01 per cent; traces only of molybdenum and earthy matter, and sulphur, by difference, 99.75 per cent. It is accordingly much more a tellurosulphur than a selenosulphur.

Concerning this tellurium, it need not be contended that it is more truly an element than that found combined with bismuth, gold, lead, and silver; it is sufficient to assert the high improbability that it should contain the same unknown elements as the latter. That being the case, then if it gives the same result by Brauner's tetrabromide method as that obtained with Hungarian tellurium, the likelihood that tellurium with atomic weight 127.6 is an element is greatly increased, if not raised to a certainty. Such was the view taken of the matter by my honoured teacher, Dr. Edward Divers, F.R.S., who placed in my hands about 14 grms. of tellurium, which had been prepared by him and Mr. Shimose years ago. They had obtained this tellurium from the sediment removed from the lead-chambers of a sulphuric acid factory, by a method the particulars of which they communicated to the *CHEMICAL NEWS* in 1883. The tellurium, which I thus received, had already been carefully freed from selenium and distilled in hydrogen.

Before I had made very much progress in preparing

For details of this method see Stas, *Mem. Acad. Belg.*, xliii., Part II., and Richards, *Proc. Amer. Acad.*, xxix., 86; xxx., 385.

for the determination of the atomic weight, a preparation which has taken a very long time, Staudenmaier's memoir came to hand, but its contents did not deter me from finishing my investigation, though they can leave no reasonable doubt, I think, that the atomic weight of the element is really 127.6.

Long as the work has occupied me, there is now no occasion to describe it in detail, since it was purposely the closest copy I could make of Brauner's operations, so far as these seemed to be material to the point. The tellurium, already so pure, was tested for impurities, and was again distilled in hydrogen.

Excellent commercial bromine was distilled from potassium bromide, zinc oxide, and water (Stas). It was dehydrated first by means of anhydrous calcium bromide left in it for some days, and then by baryta, from which it was filtered through asbestos in vessels closed from the air. It was then distilled into a receiver sealed on to the distilling flask.

The silver was first precipitated by Stas's well-known sulphite method, fused under borax and nitre, then kept for a time in fusing potassium-sodium carbonate, washed with water, hydrochloric acid, and ammonia, melted again in a lime crucible, and granulated in distilled water.

The distilled water of the laboratory was fractionally re-distilled, and the nitric acid was treated in the same way.

The balance used is one by Sartorius (his first quality), which has been hitherto only sparingly used for special cases. The weights are of quartz and platinum, from Gerhardt, and were found by me to have been closely adjusted.

The tellurium bromide was prepared by adding the tellurium to the bromine, in a tube, exactly as described by Brauner. In such a tube he directly sublimed it, but I had to transfer it to another longer tube. The procedure was to slide into this tube, nearly to the bottom, an open tube loosely fitting it, down this to drop the powdery crude tetrabromide, and then withdraw it, leaving the walls of the sublimation tube unsoiled. This tube, at once closed by a cork, was then contracted about 25 c.m. from its closed end, and again about 12 c.m. further off, where it was cut off from the corked end, and the narrowed mouth attached by caoutchouc tubing to the drying tube connected with a Sprengel pump. The tube was placed in the furnace with its first contraction just outside; the bromide before sublimation occupied the hinder third of the tube within the furnace. Sublimation was in all other respects effected just as described by Brauner, a little dibromide being sublimed off at 200° into the outer part of the tube, and the tetrabromide sublimed at a temperature kept closely at 300° into the anterior part of the tube within the furnace. Practically nothing remained unsublimed, which showed that the transference of the undistilled bromide from tube to tube had been effected with impunity, this compound not being noticeably hygroscopic, and the air, at the time, being cold and very dry. The sublimation furnace was an exact copy of Brauner's.

The tellurium bromide was weighed off and dissolved in tartaric acid in one vessel, added to the silver nitrate, shaken for hours in the bottle, with a conical, polished, pointed stopper projecting into it, and then finished off volumetrically, all just as described by Brauner (except that the final titration was not effected in a dark room, but in feeble daylight).

I made only the three determinations here given, neglecting a trial for practice, with good result, in which high accuracy was not sought for. The following are the results:—

Expt.	Tellm. brom.	Silver.	At. wt.
I.	4.1812	4.0348	127.57
II.	4.3059	4.1547	127.61
III.	4.5929	4.4319	127.58

The details of Exp. II. are:—Silver weighed off,

4.1548 grms.; time of continuous shaking by water-motor, 4 hours; precipitate, thoroughly pulverulent; silver solution added, 0.4 c.c., which produced no turbidity; potassium bromide solution required, 0.8 c.c. = 0.54 c.c. silver solution. Since there had been taken silver in excess, equivalent to 0.14 c.c. silver solution, and therefore 0.00014 grm. silver, the actual quantity of silver required by the 4.3059 grms. bromide had been 4.1547 grms. Then—

$$\frac{79.963 \times 4.1547 \times 100}{107.938 \times 4.3059} = 71.48 \text{ per cent bromine,}$$

and—

$$4 \left(\frac{107.938 \times 4.3059}{4.1547} - 79.963 \right) = 127.61 \text{ at. wt. tellm.}$$

In Exp. I, the silver weighed out was not so closely apportioned, and several c.c. of the volumetric solution had to be used; otherwise it agreed with II., as did also III. in its details.

When it is considered that Brauner and I have obtained by the same method identical results, although he worked with tellurium that had presented itself in combination with metals, while I have worked with that occurring in native sulphur of high purity, except for the presence of this tellurium and of selenium, so far as can be ascertained by tests for known elements; and when it is further considered that Staudenmaier's results are the same as Brauner's, though obtained by a wholly different method, no reasonable doubt can remain that the atomic weight of tellurium is 127.6.

The occurrence of tellurium in Japan in association with selenium in native sulphur is also a fact of great significance in settling the place of this substance in a natural classification of the elements, showing, as this does, so close a habitude to exist between it and sulphur and selenium.—*Journal of the College of Science, Imperial University, Japan*, vol. ix., Part II., p. 123.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, March 18th, 1897.

Mr. A. G. VERNON HARCOURT, President, in the Chair.

(Concluded from p. 164).

47. "Note on a Method for Determining Melting-points." By ERNEST H. COOK, D.Sc.

So many methods have been introduced for the determination of melting-points that an apology is perhaps necessary for describing another; but the following method has been found to work so well in this laboratory, and to be so easy of manipulation, that the author ventures to place it on record.

Notwithstanding, however, the theoretical simplicity of taking a melting-point, it is surprising that in commercial work considerable differences frequently occur between analysts when reporting upon such a substance, for example, as paraffin scale. Probably most, if not all, of these differences are caused by the different methods employed. Thus it is well known that "the English test," which consists in allowing the wax to solidify in a test-tube in which the thermometer is placed, gives results from 2½ to 3° Fahr. lower than the "American test," in which the wax was melted in an open dish. Both these methods again differ slightly from the capillary-tube plan, and in this process a different result is obtained when an open tube is used than when it is closed. There are, in fact, many precautions which are necessary to be observed if concordant results are to be obtained, and it is much to be desired that some distinct and definite regulations should be made with reference to the matter.

The apparatus employed is a beaker filled to the brim

with water; inside this, and separated from it on all sides, is a smaller one. The smaller beaker is partly filled with mercury in which is placed a thermometer. A stirrer is used to keep the water in the large beaker of uniform temperature. A cardboard or other disc covers the smaller beaker when the operation is in progress. The whole is heated from below by means of a sand-bath. When the melting-point to be determined is under 30° it is better to replace the sand-bath by an evaporating dish containing water.

The process is conducted as follows:—The material whose melting-point is to be taken is placed on three or four small pieces of thin ferro-type plate, or other thin metallic sheet, or on the cover glasses which are used for microscope slides. If ferro-type or other metallic slips are used, care must be taken to remove the varnish or other coating, in order that good metallic contact can be had with the mercury. The slips, with the substance on them, are now placed on the surface of the mercury, and the heat applied until the substance melts. The solidifying-point is obtained by raising the temperature above the melting-point, and allowing the beaker to cool, noting the thermometer when the first solidification takes place.

For temperatures between 100 and 200° , the larger beaker is filled with paraffin wax.

The following precautions have been found to be necessary:—(1) The temperature must be made to rise very slowly. (2) The liquid in the outer beaker must be frequently stirred. (3) Not less than $2\frac{1}{2}$ c.m. in depth of mercury must cover the inner beaker. (4) Sufficient volume of water must be allowed between the two beakers. The minimum distances to give good results are 1 inch between them laterally and $1\frac{1}{4}$ inches at the bottom. (5) The inner beaker must be immersed a sufficient depth in the water. This point is of great importance, the least distance between the top of the mercury and the top of the water being 3 inches. A greater distance is, however, to be preferred. (6) The whole apparatus should be protected from draughts. (7) The disc should be kept on the smaller beaker during the determination.

The following examples will show the degree of accuracy to be obtained in ordinary working, some of the results being obtained by students who have never taken a melting-point determination before:—Paraffin wax (1), $49^{\circ}8$, $49^{\circ}7$, $49^{\circ}5$, $49^{\circ}8$. Paraffin wax (2), $46^{\circ}2$, $46^{\circ}0$, $46^{\circ}0$, $46^{\circ}0$. Paraffin wax (3), $46^{\circ}5$, $46^{\circ}3$, $46^{\circ}5$. Ortho-mono-nitro-phenol, $44^{\circ}5$, $44^{\circ}7$. Urea, $131^{\circ}0$, $131^{\circ}5$, $131^{\circ}2$.

48. "Velocity of Urea Formation in Aqueous Alcohol." By JAMES WALKER, D.Sc., and SYDNEY A. KAY, B.Sc.

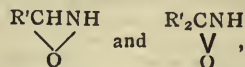
The authors have investigated the rate of formation of urea from ammonium cyanate in pure water, and in mixtures of water and alcohol, containing 10, 30, 50, 70, and 90 per cent by volume of the latter. The alcohol acts in two ways: first, it diminishes the degree of dissociation of the cyanate, and thus retards the action by diminishing the number of active molecules; secondly, it increases the rate at which the ions produced by the dissociation interact. The second mode of action outweighs the first, so that there is on the whole a marked acceleration as the water of the solvent is replaced by alcohol. If the reverse transformation of urea into cyanate, and the degree of dissociation of the latter at the various stages of the process, are taken into consideration, the requirements of the law of mass-action are strictly fulfilled.

Methylic alcohol, acetone, glycol, glycerol, and cane-sugar exert a similar accelerating effect when part of the water used as solvent is replaced by them.

From the displacement of the point of equilibrium between cyanate and urea by change of temperature, it is calculated that the transformation of ammonium ions and cyanic ions into urea is accompanied by a heat evolution of about 5000 cal. per grm.-molecule.

49. "Action of Alkyl Haloids on Aldoximes and Ketoximes." By WYNDHAM R. DUNSTAN, F.R.S., and ERNEST GOULDING.

The authors find that, when formaldoxime, acetaldoxime, and acetoxime are heated in alcoholic solution with an alkyl iodide or bromide, they are converted into compounds of alkylloximes in which the alkyl group is united to nitrogen $R'CHN(R'O)$ and $R_2CNCH(R'O)$. These derivatives are isomerides of the little-known ethers of the oximes $R'CH:NOR'$ and R_2CNOR' , and are to be regarded as derivatives of the tautomeric or isoximido-forms of the ordinary aldoxime or ketoxime—



in which the alkyl replaces the hydrogen of the amido-group. Their constitution has been proved by their hydrolysis into β -substituted hydroxylamines, $NH(R')OH$, and the corresponding aldehyde or ketone.

Formaldoxime, when mixed with methyl iodide, either in alcoholic or ethereal solution, is converted into a crystalline salt of the formula $(CH_2NOH)_3CH_3I$. It has been previously shown (Dunstan and Bossi, *Proc.*, 1894, x., 55) that formaldoxime forms salts with monobasic acids which contain 3 mols. of the oxime $(CH_2NOH)_3HCl$, &c. On hydrolysis, followed by reduction, 1 molecule of methylamine hydrochloride and 2 mols. of ammonium chloride are produced, and on heating near its melting-point (102°) only 2 molecules of formaldoxime distil from it. The formula of the compound may therefore be written $(CH_2NOH)_2 \cdot CH_2N(CH_3)O.HI$. The base corresponding with this salt could not be separated. Methyl bromide heated with formaldoxime furnishes the corresponding hydrobromide.

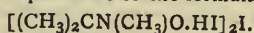
Acetaldoxime combines with methyl iodide, forming the hydriodide of a base which has so far only been obtained in the liquid state even after a process of fractional precipitation of an alcoholic solution by ether. On hydrolysis this salt furnishes acetaldehyde and β -methyl-hydroxylamine. There can therefore be no doubt that its formula is $CH_3CHN(CH_3)O.HI$. Methyl bromide combines in the same manner, forming the corresponding hydrobromide. Ethyl iodide forms the hydriodide of the ethyl derivative, $CH_3CHN(C_2H_5)O.HI$. Neither of the salts has been crystallised, and the corresponding bases are highly unstable.

Acetoxime.—By heating acetoxime with methyl iodide a red liquid is obtained, which, on concentration, deposits red crystals with a fine green lustre. The mother-liquor furnished the little-known methylamine hydriodide—



in glistening, crystalline plates (from alcohol and ether). This is a very stable non-deliquescent salt, melting at 220° with partial decomposition.

The red crystals were proved by analysis to be a methylacetoxime periodide of the formula—



On hydrolysis it breaks up into acetone and β -methyl-hydroxylamine.

Many attempts were made to isolate the hydriodide from the periodide, and also to prepare other salts from this compound, including the base, but without success, owing to the great instability of these substances.

The hydrobromide appears to be formed when methyl bromide is heated with an alcoholic solution of acetoxime, but this salt could not be crystallised.

THE FRENCH ACADEMY OF SCIENCES.

The following communication from their Correspondent in Paris appeared in *The Times* of April 7th:—

M. Berthelot read this afternoon, at the Academy of Sciences, the following letter addressed to him in French by Mr. H. Wilde, President of the Manchester Literary and Philosophical Society, announcing to the Academy the gift of £5500 to be set apart for an annual prize of

4000 frs. I send you the original letter, without undertaking, considering its special and technical character, to translate it.

"Diverses considérations m'engagent actuellement à me mettre en communication avec l'Académie dans le but de stimuler de nouvelles investigations dans les sciences physico-chimiques, et de faire disparaître quelques-uns des obstacles qui entravent leurs progrès. L'un de ces obstacles qui appelle la sérieuse attention des penseurs philosophes est l'invasion d'une autorité dogmatique dans une science scolastique, pour soutenir des erreurs démontrées et des méthodes erronées d'observation et d'expérience. Il sera suffisant pour l'objet que j'ai actuellement en vue de citer le système périodique des éléments chimiques comme un exemple de l'abus d'autorité dans une branche de la science où vous occupez un rang si distingué. J'ai à vous exprimer mes regrets que vos vues au sujet de la prétendue loi périodique ne soient venues que récemment à ma connaissance; sans cela je m'y serais référé dans mes travaux généraux sur les relations numériques des poids atomiques. Quoique vous ayez clairement indiqué, monsieur, dans vos 'Origines de l'Alchimie,' les sophismes et les contradictions inhérents à ce système, et que vous ayez également montré que la prédiction de l'existence et des propriétés des éléments inconnus n'a aucune relation nécessaire avec la prétendue loi périodique, cependant ce système a depuis été imposé aux personnes qui s'occupent de science par les sociétés scientifiques et les corps enseignants comme une vérité naturelle d'une autorité indiscutable.

"Je n'ai pas besoin de vous rappeler que l'état actuel de la chimie théorique en raison de la connaissance formelle de ce dogme est réellement déplorable. Les savants qui aspirent à se distinguer dans la chimie et dans la physique estiment qu'il est nécessaire de donner des preuves de leur croyance personnelle, en tâchant de montrer la corrélation de leurs propres travaux sur des points particuliers avec le système périodique, et ils évitent toute référence aux proportions multiples des poids atomiques, comme à une dangereuse hérésie. Beaucoup de ces néophytes, de même que certains auteurs de manuels, ne peuvent se faire une idée, ou ignorent la signification de l'idée de la périodicité telle qu'elle est définie par De Chancourtois, Newlands et Mendeleïf dans leurs mémoires respectifs. Ils appliquent l'expression impropre de loi périodique à la progression de propriétés antérieurement connues observables dans les familles naturelles des éléments, à la corrélation avec les poids atomiques de propriétés physiques et chimiques établies depuis longtemps, à la progression bien connue des propriétés physiques dans les séries homologues des composés organiques. Par suite, le danger pour les progrès future de la chimie théorique est que, lorsque l'idée illusoire d'une spiro-périodicité des propriétés analogues des éléments sera universellement abandonnée, le nom impropre de loi périodique est exposé à prendre dans la science un caractère parasite de la même façon que cette autre expression impropre, 'esprit lunatique,' avec ses dérivés, subsiste encore dans la civilisation moderne comme une survivance de la physiologie mentale barbare des âges passés.

"Heureusement pour l'avenir de la philosophie chimique que l'esprit de Dumas vit encore dans les esprits de la plupart des chimistes français, qui ne reconnaissent aucune autre autorité que la vérité de la nature telle qu'elle se présente à l'entendement, et qu'ils sont par là exempts de l'illusion de la prétendue loi périodique. En reconnaissance des nombreux profits que j'ai retirés de la science française, tant pure qu'appliquée, j'ai l'honneur d'offrir à l'Académie la somme de £5500 (137,500 f.) pour être placée en rente française, et l'intérêt provenant de cette somme devra être appliqué à la fondation d'une prix de 4000 f. à décerner tous les ans à l'auteur d'une découverte ou d'un ouvrage quelconque en astronomie, physique, chimie, minéralogie,

géologie, et mécanique, qui, au jugement de l'Académie, sera jugé le plus méritant. L'attribution de ce prix sera internationale et pourra être retrospective.

"Alderley Edge, Cheshire, 15 Mars, 1897."

The gift has given great satisfaction at the Academy, and is as much to the honour of the donor as to that of the distinguished secretary of that Academy, whose work is referred to in such terms of gratitude.

CORRESPONDENCE.

THE CHEMICAL SOCIETY ELECTION.

To the Editor of the Chemical News.

SIR,—In view of the misleading reference recently made in the press to an unfortunate incident in the celebrated Edison-Swan patent action, and as this incident has, I believe, been twisted into an argument against voting for Professor Dewar in the ill-considered contest to which he has just been exposed, I would request, Sir, that you will give publication to the answers I have received from several very eminent gentlemen who were engaged in the case, whose opinion on the incident I ventured to solicit in the interests of truth and fairness. I would beg that you will also give publication to a letter on the same subject which Mr. Crookes has received from the Right Hon. Lord Davey. I need scarcely add that the full authority of the writers has been secured for the publication of the correspondence.—I am, &c.,

HENRY E. ARMSTRONG.

86, Brook Street, W.,
March 31, 1897.

DEAR PROFESSOR CROOKES,

I was away from home yesterday, and am truly sorry that I did not get your letter in time to reply to it last night. I am afraid this letter will be of no use to you.

The incident in question occurred during my cross-examination of Professor Dewar. My recollection of it is sufficiently clear to enable me to say that I did not think that Professor Dewar intended to mislead either his Court or myself. I feel sure that the misunderstanding was due to imperfect appreciation, by the learned Judge and by myself, of the nature and object of the experiment which Professor Dewar was explaining. You may show this letter to Professor Dewar himself or anybody else.

Yours very truly,

DAVEY.

Wm. Crookes, Esq., &c., &c.

57, Onslow Square, S.W.,
March 27th, 1897.

DEAR PROFESSOR ARMSTRONG,

You have my full authority for saying that the remark made by Mr. Justice Kay about Professor Dewar's evidence in the Edison case was quite without cause. It arose, I think, from a misunderstanding on the part of the Judge as to the meaning of the evidence.

The further evidence fully substantiated the position taken by Dewar in the matter, and the Court of Appeal reversed Kay's decision.

Yours very sincerely,

J. FLETCHER MOULTON.

2, Pump Court, Temple, E.C.,
31 March, 1897.

DEAR PROFESSOR ARMSTRONG,

I am in receipt of your letter of the 29th with enclosures.

The attack upon Professor Dewar is most unfair and unjustifiable. I am intimately acquainted with every-

thing that happened in the case before Mr. Justice Kay which is referred to. No experiment of any kind was performed which could be in any way characterised as an attempt to mislead. The late Lord Justice, then Mr. Justice Kay, was mistaken, and did not understand the nature of the experiment to which he was referring. His decision was reversed in the Court of Appeal, and I say, without the slightest hesitation, that everything which Professor Dewar did upon that occasion was perfectly straightforward and justifiable. Make any use you like of this letter.

Yours faithfully,

RICHARD WEBSTER.

Holmwood, Wimbledon Common, S.W.,
31 March.

MY DEAR ARMSTRONG,

With regard to the incident in the Edison and Swan case some years ago, I thought strongly at the time, and I still think, that Dewar's answers did not tend to mislead the Court, and that he certainly did not mean to mislead.

I followed Dewar in the box, but I do not remember that I confirmed the particular experiment in question. I considered the experiment as of little consequence in the case.

Yours very truly,

J. HOPKINSON.

Athenæum Club,
April 2nd, 1897.

DEAR ARMSTRONG,

In reply to your enquiry *re* Edison and Swan *v.* Holland we have to say that, being on the opposite side to Prof. Dewar, no one could have been more likely than ourselves to take an adverse view of the experiment which formed the subject of Mr. Justice Kay's remarks.

We, however, were both of opinion that the experiment brought forward by Prof. Dewar was perfectly *bond fide*, and that there was nothing in his description of it to justify the adverse remarks of the Judge, and certainly nothing to justify the conclusion that the Judge seemed to draw from it.

We need scarcely add that there was nothing whatever in the incident which reflected upon the honesty and truthfulness of the conclusion which Prof. Dewar drew from his experiment. The garbled version which appeared in one or two journals has no doubt misled the public, both scientific and otherwise, as to the severe comments made by Mr. Justice Kay upon Prof. Dewar's experiment. The misunderstanding was completely cleared up by the evidence of subsequent witnesses and by experiments tried before Prof. Stokes on behalf of the Court.

We are

Yours very truly,

E. FRANKLAND.
WILLIAM CROOKES.

THE CHEMICAL SOCIETY ELECTION.

To the Editor of the Chemical News.

SIR,—As I had written privately to Professor Armstrong, assuring him that Dr. Collie's statement that my nomination to the Presidency of the Chemical Society was made without my knowledge or consent, I had hoped that his honourable feelings would have led him to retract publicly his statement challenging Dr. Collie's veracity. As my hopes are disappointed, I have no choice but to ask you to publish this letter, and to state that Dr. Collie's remarks at the meeting of the Chemical Society, as reported in the *Proceedings* on the 18th March, were literally true.—I am, &c.,

WILLIAM RAMSAY.

University College, London, W.C.,
April 3, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 11, March 15, 1897.

New Apparatus for the Application of Spectral Analysis to the Recognition of Gases.—M. Berthelot.—The experiments described throw a clear light on the nature of the nitrogenous principles which are formed under the influence of the effluve acting upon organic compounds. What brightens the interest is analogy of this order of reactions with those exerted between the nitrogen of the atmosphere and the immediate principles of plants.

Action of High Temperatures upon Antimony Peroxide.—H. Baubigny.—Experiment proves that antimonious acid, Sb_2O_3 , is stable at 357° ; it begins to decompose at 440° , though very slowly, and even at nascent redness the decomposition is very slow. It is not until about 750° — 800° that the antimonious acid is quickly transformed into antimony peroxide, Sb_2O_5 , which is stable at this temperature. But if it is further heated the hypoantimonious acid is decomposed in turn, perhaps at the temperature of melting silver, but assuredly a little above, and certainly below the fusion-point of gold, at which its decomposition into oxygen and volatile antimonious acid becomes fairly rapid. Antimony peroxide cannot therefore be considered as a fixed body, since it is decomposed by the mere action of heat.

Action of Tannin and other Organic Derivatives upon certain Compound Alkaloids and Ureas.—Oechsner de Coninck.—The author has experimented with pure tannin, a mixture of tannin and piperidine, gallic acid, pyrogallol, pyrocatechine, hydroquinone, and the compound ureas.

Certain Derivatives of Anethol.—Georges Darzens.—The substitution of chlorine for hydrogen in the lateral chain does not modify the odour of anethol. Carbon tetrachloride has the property of dissolving almost all organic bodies.

Fixation of Iodine by Wheat and Rice Starches.—G. Rouvier.

Solubility of the Red Pigment of the Grape and the Sterilisation of the Musts of Fruit.—A. Rosenstiehl.—The exclusion of the air is necessary for preserving the red colour of the grape and of other fruits. Musts preserved from contact with the air retain the agreeable taste of fresh grapes. The red colouring matter of the skins of the grape and of other fruits is soluble in the unfermented juice. The action of the air renders the colouring matter insoluble. It is one of the causes of the boiled taste. We may make preserves of musts, possessing the colour, the flavour, and the aroma of the fresh fruit.

No. 12, March 22, 1897.

Note on an Electric Commutator which can be Managed from a Distance.—C. Gros.

On Autoradioscopy.—Foveau de Courmelle.

Researches on the Monazite Sands.—G. Urbain and E. Badischowsky.—Will be inserted in full.

A Reaction of Carbon Monoxide.—A. Mermet.—Will be inserted in full.

On Isolauronic Acid.—G. Blanc.—The author has in a former paper described certain derivatives of isolauronic acid, especially the aldehyd $\text{C}_9\text{H}_{14}\text{O}$.

New Method of Staining Acetylene.—Georges Claude and Albert Hass.—The authors describe an experiment which consists in maintaining indefinitely in acetone at a pressure of 3 atmospheres a platinum wire heated to bright redness by the electric current.

MISCELLANEOUS.

Carbohydrates remaining in Beer. — P. Petit. — As regards inversion of acids, the dextrine of beer behaves in a manner quite different from ordinary dextrines, and its manner of inversion approximates rather to that of melitriose. — *Comptes Rendus* cxxiv., No. 10.

Royal Institution. — The following are the Lecture arrangements after Easter: — Dr. Tempest Anderson, four lectures on "Volcanoes" (the Tyndall Lectures); Dr. Ernest H. Starling, three lectures on "The Heart and its Work"; the Rev. Canon Ainger, four lectures on "Some Leaders in the Poetic Revival of 1760-1820—Cowper, Burns, Wordsworth, Scott"; Professor Dewar, three lectures on "Liquid Air as an Agent of Research"; the Reverend J. P. Mahaffy, three lectures on "The Greek Theatre according to Recent Discoveries"; Mr. J. A. Fuller Maitland, four lectures on "Music in England during the Reign of Queen Victoria" (with musical illustrations). The Friday Evening Meetings will be resumed on April 30th, when a discourse will be given by Professor J. J. Thomson on "Cathode Rays"; succeeding discourses will probably be given by "Anthony Hope," Professor Harold Dixon, the Right Hon. Lord Kelvin, Professor H. Moissan, Mr. W. H. Preece, Mr. William Crookes, and other gentlemen.

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1951.

ECONOMIC PREPARATION OF HYDROXYLAMINE SULPHATE.

By Prof. EDWARD DIVERS, M.D., F.R.S., and
TAMEMASA HAGA, F.C.S., Rigakuhakushi,
Late Asst. Prof. College of Science, Imperial University.

IN 1887 Raschig made known that hydroxylamine can be got from a nitrite by sulphonation followed by hydrolysis, and took out patents for its manufacture in this way. As to what extent these patents may have since been worked, and with what success, we have no information; but we cannot believe that this process has been advantageously carried out without great modification of the directions given. The one we are about to describe is very productive and economical for the preparation of hydroxylamine sulphate, a non-deliquescent salt, readily forming large crystals, and soluble in three-quarters of its weight of water at 20°.

Commercial sodium nitrite of 95 per cent purity does not contain more than 1 per cent of objectionable matters, such as chloride and nitrate, and is therefore pure enough. A concentrated solution of this salt (2 mols.) and of sodium carbonate (1 mol.), pretty closely adjusted in their proportions, is treated with sulphur dioxide till just acid, while it is kept well agitated at 2—3° below zero by immersion in ice and brine. At this temperature the conversion of the nitrite into oximidosulphonate is apparently perfect. Gently warmed with a few drops of sulphuric acid the oximidosulphonate rapidly hydrolyses, with marked rise of temperature, into oxyamidodisulphonate and acid sulphate. The solution of these salts is kept at 90—95° for two days, by the end of which time all oxyamidodisulphonate will have hydrolysed into hydroxylamine sulphate and sodium acid sulphate, while so small a quantity of ammonium salt is produced as can only be detected in the very last mother-liquors of crystallisation by chloroplatinic acid (potassium hydroxide being an unsuitable reagent in presence of hydroxylamine). At 80—85° five days are necessary, but then practically no ammonia is formed. At 70°, three weeks at least are necessary, while at the common temperature much oxyamidodisulphonic acid remains after several months, even when much sulphuric acid has been added. On the other hand, the solution kept boiling needs seven or eight hours usually to deprive it of all sulphonate; but the boiling has disastrous effects on the hydroxylamine, destroying at least one-third of it, by converting it (through amidodisulphonic acid?) into ammonia, and wasting another third as a practically inseparable mixture of its sulphate with ammonium sulphate.

To be assured of the disappearance of all sulphonate it is well to add barium chloride in excess to a little of the solution, and filter, and then boil the filtrate with potassium chlorate, which will change any sulphonate into sulphate. Sulphonation complete, the solution is neutralised with sodium carbonate, using methyl orange as indicator, and evaporated till it weighs only 10½ to 11 times as much as the sodium nitrite taken. Left to cool where its temperature will fall to 0° or lower, nearly all its sodium sulphate will crystallise out. The mother-liquor, evaporated sufficiently and cooled to the common temperature, yields much hydroxylamine sulphate, the mother-liquor from which, very slightly diluted and cooled below 0°, gives again a little sodium sulphate, and can be worked for more hydroxylamine sulphate, as before.

The crude hydroxylamine sulphate weighs about 9 parts for every 10 parts of sodium nitrite taken. It needs to be re-crystallised, but the mother-liquors can be closely worked up. On the other hand, the sodium sulphate re-crystallised, or even washed with ice-water, will give up 1 part more of hydroxylamine sulphate; so that sodium nitrite will yield, on the small scale, nearly its own weight of pure hydroxylamine sulphate. No doubt, on the large scale, the theoretical yield of 118·84 per cent could be more nearly approached.

Potassium nitrite is not well fitted for the preparation of hydroxylamine, because of the difficulty experienced in closely separating its sulphate from that of potassium. After several re-crystallisations the hydroxylamine salt contains 1·8 per cent of potassium sulphate. The addition of aluminium sulphate is not an improvement, for then the hydroxylamine sulphate, separated as far as practicable from the potassium alum, leaves behind on ignition as much as 5·7 per cent residue.—*Journal of the College of Science, Imperial University, Japan*, vol. ix., Part II., p. 291.

RESEARCHES ON MONAZITIC SANDS.

By G. URBAIN and E. BUDISCHOVSKY.

WE have undertaken this enquiry to find if it is legitimate to admit the existence in the monazitic sands of a new earth; the atomic weight would be approximately = 100.

In very precise and minute researches on the fractionation earths of the yttrium series obtained from the monazitic sands, P. Schützenberger and O. Boudouard have succeeded in isolating portions which cannot be split up, presenting a character of great stability, and having an atomic weight close upon 102.

More recently Drossbach (*Berichte*, xxx., 2452), on studying a monazite, arrived at similar conclusions.

In contradiction with these results, Mr. Crookes (*CHEMICAL NEWS*, No. 1931, p. 259), having examined a specimen of the yttrium earths derived from the monazitic sands, and known as *lucium*, concludes from the spectrum analysis—notwithstanding the hesitations of Schützenberger and Boudouard—that these oxides ought to be regarded as impure yttrium.

The hypothetical element supposed to be contained in the portion of the yttrium earths precipitable by sodium hyposulphite, we shall confine ourselves in this paper to a description of the experiments which we have made on the portions of the earth precipitated by this reagent, as Mr. Crookes has demonstrated that it is a very general agent of fractionation, and that it perfectly precipitates yttrium.

The sands which we studied are the same as those which have been the subject of the beautiful researches of Schützenberger and Boudouard. We repeated the treatment of the earths with potassium sulphate until the concentrated solutions—on examination with the microscope in a concentrated solution and in a stratum of 20 to 30 c.m. in depth—no longer displayed the absorption-spectrum of didymium. The absorption-spectrum of the solution free from didymium is the following:—

	Wave-lengths.
A weakening	656—649
A still weaker band	583—670
A narrow weak band	541 —
A broad band	535—517
Very faint	535—526
Very strong maximum	— 522
A very faint band	493—484

This spectrum, which is of little intensity, coincides very closely with the spectrum described for erbium.

One of us having observed that the acetylacetonates

of the rare earths are soluble in most of the organic solvents and easily admit of fractionations, it seemed interesting to apply this method to those earths, to trace if we should reach results different from those of the authors cited.

We followed very closely the course of the fractionations by the determination of the atomic weights.

The method which we employed for this does not differ sensibly from that of Schützenberger and Boudouard. Thanks to the advice of Friedel we fixed upon a method of operating which seems to us out of the reach of criticism. The salts we converted into nitrates; the solution of the nitrates is mixed with sulphuric acid, and the sulphates are evaporated to dryness to expel the chief part of the free acid. The solid sulphates are introduced into small tubes, which are heated in the vapour of mercury, by means of a bottle of mercury fitted with a reflux pipe and three muffles supported vertically. We cause the sulphates to pass successively into the different muffles, weighing them from time to time until we obtain a weight constant to about $\frac{1}{2}$ m.grm. The sulphates are then transformed into oxides, by heating them in a double platinum crucible in the Fourquignon furnace until the weight is constant. The atomic weight is easily deduced from the transformation of the sulphates into oxides. We have satisfied ourselves that on heating the oxides successively in a current of oxygen, and in a current of hydrogen, they do not undergo a perceptible variation of weight.

We prepare the acetylacetates as follows:—

The nitrates in a very dilute aqueous solution (5 grms. per litre) are precipitated with ammonia. We wash by decantation, and add the quantity of acetylacetone theoretically sufficient to transform the hydrates into crystalline acetylacetates. We filter; the crystals are fractionated at first in alcohol and then in benzene. These two reagents dissolve the acetylacetates freely in heat, and on cooling re-deposit them in needles.

In alcohol the substances of low atomic weight are concentrated in the first crystals; after six fractionations the mother-liquors and the crystals have sensibly the same atomic weights.

Collecting the portions of adjacent atomic weights we made a series of fractionations in benzene.

These results are interesting to compare with those obtained by Schützenberger and Boudouard.—*Comptes Rendus*, cxxiv., p. 618.

THE VOLUMETRIC DETERMINATION OF ZINC BY POTASSIUM FERROCYANIDE.

By L. L. DE KONINCK and EUG. PROST.

THE volumetric determination of zinc, since the publication of the Schaffner process in 1856, has formed the subject of numerous researches.

Of all the methods proposed, two only seem admissible in current practice.

On the European continent the process of Schaffner seems to be almost exclusively in use. It consists in the use of sodium sulphide. In America the Galletti procedure, as modified by Fahlberg, meets with great favour. It depends on the precipitation of zinc by potassium ferrocyanide in an acid solution (CHEM. NEWS, lxvii., 5). The ferrocyanide process appears to have been less carefully studied than that of Schaffner. It is applied in three modifications:—1. In an acid solution, as originally proposed by Galletti. 2. In a simple ammoniacal solution (A. Renard). 3. In a tartaric-ammoniacal solution. The authors confine their attention to the original procedure of Galletti. They examine the influence of time, and find that an excess of ferrocyanide corresponding to 20 per cent is sufficient to produce the transformation in fifteen minutes.

The order in which the solutions are mixed is without influence upon the result. Ammonium chloride promotes the precipitation. Ammonium nitrate has no action. To obtain very exact results the solutions must have a constant degree of acidity.

If the solution of zinc is treated with hydrogen sulphide, in order to throw down copper, cadmium, &c., it is necessary to re-oxidise the salts of iron by nitric acid or bromine.

The presence of bromine has no influence. The effects of nitric acid may be annulled by means of sodium sulphite. Manganese, if present, must be completely eliminated before titration.

All metals capable of reacting with ferrocyanide under the circumstances of the experiment must also be eliminated.

To the ammoniacal solution finally obtained, containing a quantity of ammoniacal compounds more or less constant, are added a few drops of sodium sulphite; the liquid is then neutralised with hydrochloric acid, and then acidulated with a constant quantity of the same acid.

To this solution is then added a measured volume of solution of ferrocyanide, constituting an excess of 20 to 25 p. c. on the quantity necessary for the exact precipitation. After digestion for at least ten to fifteen minutes, the quantity of zinc corresponding to the excess of the reagent is ascertained by titrating back by means of a neutral or very slightly acid solution of ZnCl_2 .

Solutions to be used.

A. A zinc solution containing per litre 10 grms. of the metal, and very slightly acid. To obtain this we dissolve 10 or 20 grms. of pure zinc in a minimum of hydrochloric acid, in a flask graduated to 1 or 2 litres with the acid of moderate heat.

When the solution is complete the liquid is brought approximately to half the final volume, and the excess of acid neutralised by means of a solution of potassium carbonate until the appearance of a slight precipitate, which is made to disappear by adding hydrochloric acid drop by drop. The liquid is brought to the ordinary temperature, and the flask filled exactly up to the mark with distilled water.

B. A solution of potassium ferrocyanide. When, as is frequently the case in technical assay, the quantity of zinc present is approximately known, we take 2 c.c. of ferrocyanide per supposed centigram of zinc, and have thus the excess of 25 per cent which we recommend to be employed.

C. The indicator is a solution of 1 per cent uranium nitrate in an aqueous solution.

Assay of Ores.

We treat a portion of the ore of 2.5 grms., dried at 100° with aqua regia if it is a blende, or with fuming hydrochloric acid if it is a calamine. When the attack is complete we evaporate to dryness to render the silica insoluble, take up the residue from the evaporation in 5 c.c. of hydrochloric acid and a little water, and then, after heating for some time to ensure the solution of the basic salts produced by evaporation, we add 50 to 60 c.c. of water and heat to 70°. We then submit the liquid to the action of a moderate current of hydrogen sulphide; during the passage of this gas we add, in several portions, 100 c.c. of water, to facilitate the subsidence of the lead and the cadmium which would not be precipitated in a solution too strongly acid. On the other hand, we must not prolong the passage of the hydrogen sulphide beyond the necessary time, nor must we dilute too strongly for fear of precipitating zinc.

The precipitate of the sulphides is collected on a filter along with the silica, if there is no reason for collecting this separately. It is acidified with 5 per cent of hydrochloric acid charged with hydrogen sulphide.

The washing is complete when the last drops of the

precipitate, rendered alkaline with ammonia, no longer give the slightest precipitate with a drop of sodium sulphide.

The filtrate is heated at ebullition until the hydrogen sulphide is expelled; it is mixed with 10 c.c. of fuming hydrochloric acid and from 10 to 25 c.c. of saturated bromine water (according to the proportion of iron), so as to re-oxidise the ferrous salts and assist the precipitation of manganese. It is then passed drop by drop, whilst constantly stirring, into a flask marked at 500 c.c., containing 100 c.c. of concentrated ammonia and 10 c.c. of solution of ammonium bicarbonate more or less saturated in the cold (about 20 to 25 per cent). It is allowed to cool, water is added up to the mark, stirring so as to render the mixture homogeneous, left for a short time to deposit the precipitate, and filtered with a dry filter.

The method described is that applicable to the preparation of the solution for the most complex ores. In the absence of metals precipitable by hydrogen sulphide the treatment with that reagent is omitted, and the re-oxidation of the iron salts is unnecessary, since they have not been reduced.

The use of bromine is needed only if the ore is maniferous.

If the ore contains metals requiring treatment with hydrogen sulphide, but no manganese, the re-oxidation of the ferrous salts may, if preferred, be effected by boiling nitric acid.

We take 100 c.c. of the ammoniacal filtrate prepared as above directed, add a few drops of sulphite, and pour in gradually hydrochloric acid (sp. gr. 1.075) until a small morsel of litmus-paper thrown into the liquid shows, in passing to redness, that the point of neutralisation has been reached (about 30 c.c.), and we then add further 10 c.c. of the same acid.

(To be continued).

A REVISION OF THE ATOMIC WEIGHT OF MAGNESIUM.*

By THEODORE WILLIAM RICHARDS
and
HARRY GEORGE PARKER.

(Concluded from p. 175).

Third Series of Determinations.

IN order to remedy the most serious defect of the second series, the arrangement for drying the air and nitrogen was much enlarged and improved. By pouring sulphuric acid into the safety funnels, at the top of the many towers, from time to time, during the passage of the gas, the glass beads were kept thoroughly saturated during the whole process. The sulphuric acid having reached the bottom of the column, drained out of the tubes provided for that purpose into beakers below. It will be seen that by this means the efficiency of the apparatus was far greater than in the previous form. As a test, a very rapid stream of wet air from a water blast was passed through the apparatus and then through a weighed phosphorus pentoxide bulb for nearly two hours, without the slightest appreciable increase of weight of the pentoxide bulb. The same test was applied to the apparatus for drying the hydrochloric acid gas, with the same result.

With the help of this important addition to the apparatus, another series of determinations was now made. The somewhat lower result of this series is undoubtedly due to the more perfect desiccation of the gases; the agreement of the individual results is still not quite perfect, but the series is undoubtedly far more reliable than the second.

* Contributions from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, vol. xxxii., No. 2.

SERIES III.

No. of expt.	Sample used.	Sample of $MgCl_2$ of Ag used.	Weight of $MgCl_2$ of Ag.	Weight of Ag.	Ratio. $MgCl_2 : 2Ag = 100 : n$.	Atomic weight of Mg.
9	1	2	1.99276	4.51554	226.597	24.349
10	1	2	1.78870	4.05256	226.565	24.363
11	1	2	2.12832	4.82174	226.551	24.369
12	2	2	2.51483	5.69714	226.542	24.373
13	2	3	2.40672	5.45294	226.571	24.360
14	2	3	1.95005	4.41747	226.531	24.377
Average						24.365

Fourth and Final Series of Determinations.

The apparatus was now put in the best possible order, and the phosphorous pentoxide tubes were re-charged, in order to make ready for a series of determinations in which the very highest exactness was to be aimed at. The purest samples of material were used, and all other precautions, learned from previous work, were taken to insure accuracy. The following determinations were consecutive, with the exception of one between Nos. 15 and 16, which was spoiled by a slight accident.

SERIES IV.

No. of expt.	Sample used.	Sample of $MgCl_2$ of Ag used.	Weight of $MgCl_2$ of Ag.	Weight of Ag.	Ratio. $MgCl_2 : 2Ag = 100 : n$.	Atomic weight of Mg.
15	2	3	2.03402	4.60855	226.573	24.360
16	2	3	1.91048	4.32841	226.562	24.364
17	2	3	2.09932	4.75635	226.566	24.362
18	2	2	1.82041	4.12447	226.568	24.362
19	2	2	1.92065	4.35151	226.565	24.363
20	3	4	1.11172	2.51876	226.564	24.363
Average						24.362
Extreme difference ..						0.004

These results agree with one another as well as could possibly be expected, for the difference between the extremes in the last series corresponds to a difference of only one-tenth of a milligram, in the weight of the magnesian chloride. Since two wholly distinct samples of this salt and three wholly distinct samples of silver were used in this series, we may conclude that all ordinary accidental errors had been eliminated; and in a critical discussion of the result we may limit ourselves to the consideration of the possible constant errors of the process.

The most serious objection to the method is, of course, the possible retention of water, of magnesian oxychloride, or of ammoniac chloride by the magnesian salt.

With regard to the first two difficulties, it need only be said that the gases used for drying the magnesian chloride were as dry as present possibilities permit them to be made. The phosphorus pentoxide in the last drying tube showed no trace of liquefaction at the close of the research, but seemed to be as light and powdery as at first, in spite of the fact that several hundred litres of gas had been passed over it. Any trace of oxygen, as well as of aqueous vapour, was excluded from the hot salt; for the hydrochloric acid gas was replaced by nitrogen, and this was driven out in its turn by dry air only after the tube had cooled. A means of proving absolutely that no water remained does not exist; but it is extremely hard to see how water could have gained access to the carefully guarded magnesian chloride.

The fact that every sample of magnesian chloride used in the last series gave an absolutely clear and transparent solution in water is additional evidence of much weight; for a very small trace of oxychloride would have shown itself in opalescence. As a proof of this it may be stated that in experiment No. 12 of Series III, there was a perceptible cloudiness upon the solution of the magnesian chloride in water, owing to a known access of a trace of aqueous vapour, caused by a momentary stoppage of the

current of nitrogen. This result is, however, scarcely at all different from the others.

With regard to the possible retention of ammoniac chloride by the magnesium salt, it may be said:—First, that none could be detected by means of a Nessler solution; and, secondly, that even if a small amount had been retained, it would have made but a very slight difference in the final result.

Our result is essentially the same, no matter whether the chlorine is weighed as argentic chloride (Series I.), or the amount of silver necessary to precipitate it is found (Series III. and IV.). This fact is satisfactory evidence that the silver and chlorine were both pure, as well as that no magnesian chloride was occluded by the argentic chloride. Thus:—

From the ratio $2\text{AgCl} : \text{MgCl}_2$ (Series I.), $\text{Mg} = 24.369$.
 " " $2\text{Ag} : \text{MgCl}_2$ (Series III.), $\text{Mg} = 24.365$.
 " " $2\text{Ag} : \text{MgCl}_2$ (Series IV.), $\text{Mg} = 24.362$.

Upon comparing these figures with the older ones, they are seen to agree surprisingly with Marignac's value obtained from work upon magnesian oxide and sulphate ($\text{Mg} = 24.37$). Burton and Vorce's syntheses of magnesian oxide gave a lower value for magnesium (24.28); but if these were corrected for a probable amount of gases in the magnesian oxide, the result would probably be close to the present one. The analytical chemist should not forget that the value 24.36 is $1\frac{1}{2}$ per cent higher than the round number 24 , which has been so commonly accepted.

For reasons which must be manifest to any careful reader of the foregoing paper, we accept the value given by the fourth and last of our series as representing the most probable atomic weight of magnesium. It remains only to state this result in terms of the usual unfortunately varying standards of reference used by the scientific world.

If $\text{O} = 16.000$, $\text{Mg} = 24.362$
 If $\text{O} = 15.96$, $\text{Mg} = 24.301$
 If $\text{O} = 15.88$, $\text{Mg} = 24.179$

THE ATOMIC WEIGHTS OF NITROGEN AND ARSENIC.*

By JOSEPH GILLINGHAM HIBBS.

THE atomic weight of the metal molybdenum had been determined by expelling molybdic acid from sodium molybdate with hydrochloric acid gas, then weighing the residual sodium chloride.

Having found that nitric acid and arsenic acid were driven from their alkali salts with ease, leaving a chloride that was absolutely pure, and believing that the atomic masses of nitrogen and arsenic determined in this manner would afford a valuable contribution to the literature relating to these constants, a carefully conducted series of experiments was made with two nitrates and one arsenate. The results are given in detail in the following lines:—

The Atomic Weight of Nitrogen.

In the past, determinations of the atomic weight of nitrogen have been made from the density of the gas itself, from the ratio between ammonium chloride and silver, and from the decomposition of certain nitrates. The first method in particular has been frequently applied. Thomson, Dulong, Berzelius, and Lavoisier brought to light many new facts relating to the atomic weight of nitrogen; unfortunately, however, they have been affected by complications that have introduced inaccuracies.

* Contribution from the John Harrison Laboratory of Chemistry. From the author's thesis presented to the faculty of the University of Pennsylvania for the degree of Doctor of Philosophy, 1896. From the *Journal of the American Chemical Society*, vol. xviii., No. 12.

Dumas and Boussingault (*Comptes Rendus*, 1841, xii., 1005) found the mean density of nitrogen to be 0.972 ; for hydrogen they found a mean density of 0.0693 , which would give nitrogen an atomic weight of 14.026 . Regnault obtained a more concordant series of results, the mean being 0.97137 , and a density for hydrogen of 0.0692 , which makes the atomic weight of nitrogen equal to 14.0244 .

Clarke gives in detail his computation of the means of the results obtained by Penny, Stas, and Marignac. Their work on the determination of the atomic weight of this particular element was mainly on the ratio of ammonium chloride and silver, and the decomposition of certain nitrates. A great degree of accuracy was maintained throughout the entire investigation; but the amount of work required to obtain a single result necessarily lays the method open to a serious error of manipulation.

In this connection a paragraph from Clarke's "A Recalculation of the Atomic Weights" may be cited:—"The general method of working upon these ratios is due to Penny. Applied to the ratio between the chloride and nitrate of potassium, it is as follows:—A weighed quantity of the chloride is introduced into a flask which is placed upon its side and connected with a receiver. An excess of pure nitric acid is added, and the transformation is gradually brought about by the aid of heat, the nitrate being brought into a weighable form. The liquid in the receiver is also evaporated, and the trace of solid matter which has been mechanically carried over is recovered and also taken into account."

The method indicated in this study, and actually applied with the results appended, is decidedly less objectionable. In this method there is no distillation, no precipitate; in fact, nothing that could involve serious error.

Clarke summarises the results of Penny, Stas, and Marignac as follows:—

1. From specific gravity of N	N = 14.0244
2. " ammonium chloride	N = 14.0336
3. " ratio number four	N = 14.0330
4. " silver nitrate	N = 13.9840
5. " potassium nitrate	N = 13.9774
6. " sodium nitrate	N = 13.9906

Mean of results for N N = 14.0210

If oxygen is 16, this becomes 14.0291 . Stas found the atomic weight of nitrogen to be 14.044 . Dumas found 14 by experiments on the combustion of ammonia and cyanogen ($\text{O} = 16$). Pelouze found 14.014 by bringing a known weight of silver nitrate in contact with a known and slightly excessive weight of ammonium chloride, which excess was titrated. Anderson found 13.95 by the decomposition of the nitrate of lead, with just enough heat for decomposition (the same method that was used by Berzelius). Marignac found 14.02 by dissolving a known weight of silver in nitric acid and then melting and weighing the nitrate found.

A.—Atomic Weight of Nitrogen by Action of Hydrogen Chloride upon Potassium Nitrate.

The purest salt obtainable was dissolved in water, filtered, and re-crystallised six times, a solution of which was tested for chlorides, sulphates, &c., but no impurity was found. One more crystallisation was made and the best crystals were selected. These were washed with distilled water and dried at 210°C . for three hours, powdered, and again dried, and finally placed in a weighing bottle. This compound was dried before each experiment. It was also allowed to stand in a balance case one hour before weighing. The same degree of care was exercised in the preparation of the boat for weighing.

The weighing bottle was placed on the scale pan and

TABLE A.

No.	Potassium nitrate taken. Grm.	Potassium chloride obtained. Grm.	Correction for potassium nitrate. Grm.	Correction for potassium chloride. Grm.	Correction for weight of potassium nitrate. Grm.	Correction for weight of potassium chloride. Grm.	Molecular weight of potassium nitrate obtained.	Atomic weight of nitrogen obtained.
1.	0'11084	0'08173	0'00006	0'00004	0'11090	0'08177	0'101121	14'011
2.	0'14864	0'00960	0'00007	0'00005	0'14871	0'10965	0'101120	14'010
3.	0'21056	0'15525	0'00011	0'00008	0'21067	0'15533	0'101123	14'013
4.	0'23248	0'17214	0'00012	0'00009	0'23360	0'17223	0'101121	14'011
5.	0'24271	0'17894	0'00013	0'00009	0'24284	0'17903	0'101124	14'014

Atomic weight of nitrogen = $14'0118 \pm 0'000472$.

TABLE B.

No.	Potassium nitrate taken. Grm.	Sodium chloride obtained. Grm.	Correction for sodium nitrate. Grm.	Correction for sodium chloride. Grm.	Correction for sodium nitrate. Grm.	Correction for sodium chloride. Grm.	Molecular weight of sodium nitrate.	Atomic weight of nitrogen.
1.	0'01550	0'01064	—	—	0'01550	0'01066	85'061	14'011
2.	0'20967	0'14419	0'00009	0'00007	0'20976	0'14426	85'061	14'011
3.	0'26217	0'18029	0'00012	0'00009	0'26229	0'18038	85'064	14'014
4.	0'66610	0'46805	0'00035	0'00024	0'66645	0'45829	85'064	14'014
5.	0'93676	0'64422	0'00042	0'00034	0'93718	0'64456	85'058	14'008

Atomic weight of nitrogen = $14'0116 \pm 0'000741$.

TABLE C.

No.	Sodium pyroarsenate taken. Grm.	Sodium chloride obtained. Grm.	Correction for sodium pyroarsenate. Grm.	Correction for sodium chloride. Grm.	Correction for sodium pyroarsenate. Grm.	Correction for sodium chloride. Grm.	Molecular weight of sodium pyroarsenate.	Atomic weight of arsenic.
1.	0'02176	0'01439	0'00001	0'00000	0'02177	0'01439	354'008	74'904
2.	0'04711	0'03114	0'00002	0'00001	0'04713	0'03115	354'042	74'921
3.	0'05792	0'03828	0'00003	0'00002	0'05795	0'03830	354'054	74'927
4.	0'40780	0'26970	0'00021	0'00011	0'40801	0'26981	354'002	74'901
5.	0'50440	0'33028	0'00026	0'00017	0'50466	0'33045	354'033	74'916
6.	0'77497	0'51222	0'00041	0'00027	0'77538	0'51249	354'034	74'917
7.	0'82853	0'54762	0'00044	0'00029	0'82897	0'54791	354'034	74'917
8.	1'19'68	0'78690	0'00056	0'00041	1'19124	0'78731	354'053	74'926
9.	1'67464	1'10681	0'00081	0'00051	1'67545	1'10732	354'057	74'928
10.	3'22485	2'13168	0'00152	0'00099	3'22637	2'13267	354'002	74'901

Atomic weight of arsenic = $74'9158 \pm 0'00222$.

allowed to stand several minutes in order to regain its normal temperature. After weighing it was quickly opened and a portion of the salt removed to the boat, and again closed and allowed to stand in the balance case for several hours before re-weighing. The boat was then introduced into the combustion tube and the gas passed over it. The characteristic action took place, The only difference in the method of procedure adopted here and that described in the first section of this paper, was a longer time being given to complete the action, using a lower temperature, in order to do away with all possibility of fusion of the salt. It was then carefully removed to a vacuum desiccator and allowed to stand over night before weighing. It may be said also that experiments were only conducted on clear days to insure the non-entrance of moisture.

With potassium nitrate, no great variation of amount was taken.

Five determinations were made in this case (Table A).

The atomic values used in these calculations were taken from "Table of Atomic Masses," revised by F. W. Clarke, in October, 1891.

The figures deduced from these values are, of course, subject to any change made by later revision of atomic weights. It is not so much the exact figure to which attention is called, as to the constancy of result brought forward by this method. The values used were:—

Oxygen	16'00
Potassium	39'11
Chlorine	35'45
Specific gravity potassium nitrate	2'1
Specific gravity potassium chloride	1'99

B.—Atomic Weight of Nitrogen by Action of Hydrogen Chloride upon Sodium Nitrate.

The same degree of care and method of procedure were here observed as in Division A. The results are given in Table B.

Atomic values used were:—

Oxygen	16'00
Sodium	23'05
Chlorine	35'45
Specific gravity sodium chloride	2'16
Specific gravity sodium nitrate	2'26

When these results are compared with those obtained by Penny and Stas by treatment of potassium chloride with nitric acid, and the treatment of potassium nitrate with hydrochloric acid (likewise for sodium), a close comparison can be made.

	Penny.	Hydrogen chloride method.
For potassium nitrate	13'9774	14'0118
For sodium nitrate ..	13'9906	14'0116

Showing a difference of—

0'0344 for potassium salt,
0'0210 for sodium salt.

When a mean of the above results is taken, the atomic weight of nitrogen equals—

13'9996 for potassium salt,
14'0011 for sodium salt.

Taking now a mean of these values, the atomic weight of nitrogen would be 14'0003.

C.—The Atomic Weight of Arsenic.

The atomic weight of arsenic has been obtained from the chloride (AsCl_3), the bromide (AsBr_3), and the trioxide (As_2O_3).

Pelouze, in 1845 (*Comptes Rendus*, x., 1047), and Dumas, in 1859, determined it by the titration with known quantities of pure silver in the analysis of arsenic trichloride. The mean of their results, as computed by Clarke, gives the atomic weight of arsenic 74·829. Wallace (*Phil. Mag.*, (4), xviii., 279) makes the same titration with silver in the analysis of arsenic tribromide. His value is 74·046. Kessler made a set of determinations by estimating the amount of potassium bichromate required to oxidise 100 parts of arsenic trioxide to arsenic pentoxide. He obtained a mean value of 75·002.

A mean of these results gives the following:—

From AsCl_3		74·829
„ AsBr_3		74·046
„ As_2O_3		75·002
General mean		74·918

If oxygen=16 then the atomic weight of arsenic will equal 75·090.

Berzelius, in 1826, heated sulphur and arsenic trioxide together in such a way that sulphur dioxide alone escaped; this method gave 74·840 as the atomic weight of arsenic. But one experiment was made, so that it does not possess much value. In the above method there seems to be a wide variation in the results obtained, the difference between the extreme values is but little less than one unit.

By the hydrogen chloride method, we have but the weighing of the material used in the determination—which must necessarily enter every estimation or analysis—and a single weighing after the action of the acid gas. As in the case of nitrogen, the method seems to be as short and concise as possible.

The methods and *modus operandi* were exactly the same as those used in the determination of the atomic weight of nitrogen.

The sodium chloride obtained was perfectly white in colour. In no instance was it fused. After weighing the salt residue it showed no traces of arsenic, and was readily soluble in cold water without residue. The same conditions of atmosphere were observed.

As the specific gravity of sodium pyroarsenate could not be obtained, it was determined by means of the specific gravity bottle, against chloroform, and was found to be 2·205, while the specific gravity of sodium chloride was taken as 2·16. The atomic values used were:—

Oxygen		16·00
Sodium		23·05
Chlorine		35·45

The results here obtained, besides being to a great degree constant, compare favourably with those obtained by Pelouze (74·829) and Kessler (75·002).

A coincidence may here be shown by the fact that the mean of these values gives 74·9155, while the hydrogen chloride method gives 74·9158.

In order to give the method a thorough trial, the amounts taken cover a wide range. The smallest amount used was 0·02176 gm. of sodium pyroarsenate, and the largest 3·22485 grms. It will also be noticed that the variation in result is but 0·027 for ten determinations (Table C).

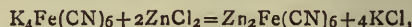
NOTES ON THE FERROCYANIDES OF ZINC AND MANGANESE.

By EDMUND H. MILLER.

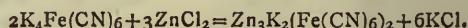
THE composition of the ferrocyanides of zinc and manganese, formed when salts of these metals are precipitated

by potassium ferrocyanide, is given by Prescott and Johnson ("Qualitative Analysis," pp. 67 and 57) as $\text{Zn}_2\text{Fe}(\text{CN})_6$ and $\text{Mn}_2\text{Fe}(\text{CN})_6$, while the books on volumetric analysis, such as Sutton's and Beringer's, ignore the composition of this precipitate.

The prevailing idea is that in the titration of zinc by potassium ferrocyanide, a normal zinc ferrocyanide is formed. This I believe to be incorrect, for if the reaction is—



a solution of potassium ferrocyanide, 1 c.c. of which is equivalent to 10 m.grms. of zinc, would contain 32·32 grms. of $\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}$ to the litre, not 43·2 (Sutton, "Volumetric Analysis," p. 329; Beringer, "Assaying," p. 219) to 45 grms. (Furman, "Assaying," p. 205), as has been found by experiment. Using 44 grms. per litre as a basis for calculation, the reaction becomes—



This reaction is not merely one that may possibly be true, but according to Wyruboff (*Ann. Chim. Phys.*, [5], viii., 485), the precipitate formed by the action of potassium ferrocyanide on a zinc salt, whichever is in excess, is $3\text{Zn}_2\text{Fe}(\text{CN})_6 \cdot \text{K}_4\text{Fe}_3(\text{CN})_6 \cdot 12\text{H}_2\text{O}$, white, while the normal salt, $\text{Zn}_2\text{Fe}(\text{CN})_6 \cdot 4\text{H}_2\text{O}$, is formed only by the action of hydroferrocyanic acid on a zinc salt.

This statement agrees both with the preceding reaction and with the results obtained in standardising potassium ferrocyanide solution.

The manganese precipitate with potassium ferrocyanide, as obtained in titration, is given by Stone (*Journ. Amer. Chem. Soc.*, xvii., 473) as $\text{Mn}_3\text{Fe}_2(\text{CN})_{12}$. This is a ferri-, not a ferrocyanide, thus making necessary a change of quantivalence. Mr. Stone also states that an amount of potassium ferrocyanide which will precipitate 4 atoms of zinc will only precipitate 3 of manganese, thus basing his calculation on the formation of normal zinc ferrocyanide.

Wyruboff (*Ann. Chim. Phys.*, [5], viii., 474) gives the precipitate obtained from potassium ferrocyanide and manganese salt, whichever is in excess, as—



while the normal salt $\text{Mn}_2\text{Fe}(\text{CN})_6 \cdot 7\text{H}_2\text{O}$, cream, is formed as in the case of zinc by hydroferrocyanic acid.

The solution used by Mr. Stone had the following strength:—

- 1 c.c. = 0·00606 gm. zinc.
- 1 c.c. = 0·00384 gm. manganese.

If the ratio were exactly four zinc to three manganese, using the most recent atomic weights, the strength of this solution against manganese would be 1 c.c. = 0·00382 gm.; while, according to Wyruboff, $10\text{Mn} = 9\text{K}_4\text{Fe}(\text{CN})_6$ and $6\text{Zn} = 4\text{K}_4\text{Fe}(\text{CN})_6$, or $10\text{Mn} = 13·5\text{Zn}$, or $1\text{Mn} = 1·35\text{Zn}$, and the strength against manganese would be 1 c.c. = 0·003774 gm.

These figures show but little difference between the two ratios, and, while Mr. Stone's experimental results are undoubtedly accurate, his theory based on the formation of $\text{Zn}_2\text{Fe}(\text{CN})_6$ and $\text{Mn}_3\text{Fe}_2(\text{CN})_{12}$ is not satisfactorily proved.

This article is only a preliminary note regarding the composition of the ferrocyanides as they are being investigated in this laboratory.

In connection with the ferrocyanide of zinc I have found a very strong solution of hydrochloroplatinic acid, H_2PtCl_6 , acidified with hydrochloric acid, a most satisfactory indicator for the titration of zinc by potassium ferrocyanide, when performed in a hot solution. This indicator is used in the same way as uranium acetate, and is less affected by a varying amount of hydrochloric acid. The end reaction is a bright emerald-green, which takes a few seconds to develop. It will not work with a cold solution.—*Journal of the American Chemical Society*, xviii., No. 12.

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, April 9th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

MR. T. A. GARRETT read a Paper on "*A Nickel Stress Telephone.*"

In conjunction with Mr. W. LUCAS, the author has experimented upon telephones with nickel magnets. A magnetised nickel rod is wound with insulated wire, and is then fixed vertically by a clamp at its lower end. A wooden diaphragm is rigidly attached to the top of the rod, in a horizontal plane. The rod just passes through the middle of the diaphragm, where it is fixed with sealing-wax. The diaphragm is entirely supported by the nickel rod. On speaking against the top of the diaphragm, variations of longitudinal pressure, and consequently of magnetisation, are produced in the nickel; and corresponding undulatory currents are induced in the surrounding coil. The nickel wire is sometimes magnetised by stroking it with a magnet, and sometimes by passing a current through the coil. A diaphragm of pine wood gives better results than a metallic plate. The instrument does not work well as a "receiver"; an ordinary telephone is used for this latter purpose. The results obtained with a weakly magnetised nickel rod are much better than those with a strongly magnetised steel rod, indicating that the undulatory currents are due rather to magnetic variations arising from changes of stress than to the relative motions of the magnet and coil.

Dr. S. P. THOMPSON said that some years ago he had worked with a somewhat similar apparatus, using it as a "receiver," with wires of nickel, cobalt, and iron. Cobalt gave the best results; the metallic strips in his experiments dipped into the solenoids without contact with them. This arrangement did not work well as a "transmitter," even when a battery was included in the circuit. In some cases the rods were cut into short lengths separated by brass.

Mr. BOYS asked how the nickel "stress" instrument compared in clearness and loudness with an ordinary telephone.

Mr. SHELFORD BIDWELL had tried a nickel telephone with a mica diaphragm; depending not upon mechanical stress, but magnetic strain. It did not work well.

Dr. CHREE thought the "stress" telephone might possibly be improved by choosing the right strength of magnetic field.

Mr. APPEYARD said the arrangement was interesting historically, because it was, mechanically, almost identical with the original instrument used by Philip Reis as a "receiver." The authors had succeeded in getting it to work as a "transmitter." Their success was probably due to the rapidity with which the magnetisation of nickel responded to very small changes of stress or current. The Post-Office electricians had tried to introduce nickel cores into relays, on account of its magnetic sensitiveness; the results, he believed, had not been very satisfactory.

Mr. T. A. GARRETT, in replying, said the "stress" telephone gave better articulation than an ordinary "watch" telephone, but the sounds were feebler. There seemed to be a field-strength proper to the instrument; he had noticed that the articulation was clearer with three cells than with six.

Mr. W. A. PRICE then read a Paper on "*Alternating Currents in Concentric Conductors.*"

This is a mathematical investigation of a proposed new form of submarine cable. The case is considered of two concentric conductors, interrupted alternately at different

points throughout the whole length. In the mathematical treatment the cable is supposed to be laid in a circular path, and successive charges of electricity are supposed to be applied at some point at the extremity of a diameter of the circle. Expressions are given for the amplitude of the periodic charges arriving at a point diametrically opposite to the first; and for the reduction in amplitude, throughout the whole length of the cable, of an applied E.M.F. The theory indicates that under no circumstances can the "speed" of a cable of the proposed form be greater than the "speed" of a cable of ordinary type. The author has experimented upon an artificial cable connected up to represent the proposed form. The "definition" of signals is considerably better than that obtained through an artificial cable of analogous "weight" and "length" connected up in the ordinary way. Within certain limits the "definition" continues to improve as the number of sections, or subdivisions, of the cable is increased.

Mr. BLAKESLEY said he was sorry the result did not indicate a successful type of cable. He would have been inclined to predict that the amplitude would have decreased with the number of sections. If a number of condensers were joined in series, and one end was subjected to a periodic E.M.F., the amplitude would fall off inversely as the square of the distance.

Mr. PRICE then exhibited a galvanometer support. The instrument is suspended from two indiarubber cords attached at the top and bottom to cross-bars of metal, thus forming a rectangle. The cross-bars are provided with knife-edges in such a way as to compensate for unequal stretching of the indiarubber. Weights can be added, if necessary, to the support, so as to increase its inertia.

Mr. H. GARRETT read a Paper, communicated by Prof. W. B. MORTON, on "*The Effect of Capacity on Stationary Electrical Waves in Wires.*"

The author investigates the effect produced when a condenser is inserted at a point in the secondary circuit of the apparatus used by Blondlot for obtaining stationary electrical waves in wires. The positions of successive nodes are determined in the usual way, by a bridge, with a vacuum-tube indicator. When two opposite points of the parallel secondary wires are joined to the plates of a small air-condenser, the nodes approach the condenser on either side. The amount of the displacement of the nodes—that is to say, the extent of the shortening of the apparent half wave-length—depends upon the position of the capacity along the wires. The effect is *nil* when the condenser is at a node, and a maximum when it is midway between two nodes. The state of affairs at a point of the circuit is obtained by summation of a series of separate disturbances due to the different direct and reflected trains. In obtaining a formula for the conditions of resonance, with which to compare the observations, the author adopts a method from Heaviside. It connects the frequency of oscillation with the position and capacity of the condenser.

Mr. SHELFORD BIDWELL proposed a vote of thanks to all the authors, and the meeting was adjourned until May 14th.

Action of Nickel upon Ethylene.—Paul Sabatier and J. B. Senderens.—The authors have caused ethylene to act upon nickel obtained by reducing the oxide with hydrogen. After cooling in a current of hydrogen the metal is exposed to ethylene which has been carefully dried and purified. There is no effect in the cold, but about 300° and more readily at a higher temperature the nickel gradually sprouts, yielding a very voluminous black matter. This matter is carbon in which nickel is distributed.—*Comptes Rendus*, cxxiv., No. 12.

NOTICES OF BOOKS.

A Detailed Course of Qualitative Chemical Analysis of Inorganic Substances. With Explanatory Notes. By ARTHUR A. NOYES, Ph.D., Assistant Professor of Chemistry in the Massachusetts Institute of Technology (Boston). Third Revised and Enlarged Edition. New York: The Macmillan Company. 1897. 89 pp., 8vo.

An Introductory Course of Quantitative Analysis. With Explanatory Notes and Stoichiometrical Problems. By HENRY P. TALBOT, Ph.D., Associate Professor of Analytical Chemistry in the Massachusetts Institute of Technology (Boston). New York: The Macmillan Company. 1897. 125 pp., 8vo.

THOSE who follow the fashion of deprecating the rapid multiplication of chemical text-books do not fully comprehend the conditions which impel the authors to issue them.

Teachers finding themselves expected to give instruction in elementary chemistry to large classes of young men having no previous experience in manipulation, and finding it impossible to give to each member of the class that personal assistance and supervision which is well nigh indispensable to success, are compelled to plan courses of laboratory work adapted to the circumstances, and to prepare, in manuscript, directions embracing such minute details that the students cannot possibly go astray. These manuscript notes grow from year to year with the needs of successive classes, and gain in value by the experience of the teacher, until after a few years the instructor finds it more economical of the time of the student to print these notes than to communicate the statements orally. Moreover, each Institution establishes courses having different ultimate aims, one being intended to qualify the students for the pursuit of mining, another for engineering, a third for the vocation of geologist or of naturalist, and, in consequence, the character of the chemical work required is modified to suit each case. This does not imply that the fundamental facts of chemistry differ in the several courses of instruction, but that the topics treated are selected to lead the students in the direction of the goals.

Again, the amount of time that can be devoted to chemical instruction is sometimes greatly abbreviated, so that the instructor finds indispensable the utmost condensation, consistent with perspicuity. Hence standard treatises on analytical chemistry, such as those of Fresenius, are relegated to the position of books of reference, and the students are supplied with specially adapted guides.

We do not claim, but we surmise, that the books under review have arisen in some such way, and this, instead of being a disadvantage, is one of the causes of their excellence.

Professor Noyes's little work has evidently found a larger circle of friends than the few students in his own classes, for it has reached a third edition. A characteristic feature of the book is the separation of the methods of procedure from the notes upon them, the former consisting of very detailed directions for carrying out the processes, and the notes serving to explain the philosophy of the same. The section on the detection of metals is followed by one on the detection of acids, and a third on analysis by the dry method. These are succeeded by instructions for the preparation of the solutions, and an appendix on the preparation of reagents.

Professor Talbot's treatise is intended for beginners who have completed a course in qualitative analysis, and follows the same method as that of Professor Noyes. The author remarks that his book should be supplemented by reference to the works of Fresenius, Mohr, and Sutton. Instead of selecting simple salts for analysis as preliminary practice, Dr. Talbot uses approximately pure samples of appropriate minerals or industrial products:

this advances the student more rapidly without greatly increasing the difficulties. Part IV., Stoichiometry, is a valuable feature.

Both Prof. Noyes and Prof. Talbot are to be congratulated on the handsome paper and typography which is furnished by Macmillan and Co., the publishers.—H. C. B.

Laboratory Manual. A Short Course of Practical Chemistry. By ALFRED C. BEEBE. Chicago: A. Flanagan.

THE work before us is a favourable specimen of those elementary treatises on chemistry in which the English press has been so prolific, and which are now also appearing in America. The author lays some emphasis upon a reaction for the recognition of potassium, which has been proposed by Koninck and approved of by W. Crookes. It has the advantages of being cheaper than platinum chloride, and more manageable than tartaric acid. Otherwise, whilst considering the instructions conveyed in this book sound and purposive, we fail to find them superior to those met with in similar manuals.

Les Nouveautés Chimiques par 1897. Nouveaux Appareils de Laboratoires, Methodes Nouvelles de Recherches appliquées à la Science et à l'Industrie. CAMILLE POULENC, Sc.D. Paris: J. B. Baillière et Fils. 1897.

THIS work has been compiled in order to give chemists and physicists an account of recent discoveries fuller and more accurate than the notices which we encounter in the literary and the political press. The first chapter is taken up with general applications of chemistry and physics, such as Dr. Joly's meldometer for the rapid and accurate determination of the fusion-points of bodies which melt at high temperatures; the pyrometric telescope of Mesuré and Nouel; the new self-correcting air-thermometer of F. G. Müller; the short thermometers of Dr. Raikow; McTear's thermo-element for determining high temperatures; the Wiborgh thermophore, for determination of elevated temperatures; the double differential thermoscope of Loesen; Dunnington's new gas-regulator; various apparatus for the production of acetylene; M. Grünberg's proposed appliance for the rapid determination of the specific gravity of ores; the universal densimeter of Courtonne; the densimeter of Pieri; the compensating densimeter of Galaine; Vandevyn's new areometer.

An important section of the book is devoted to bacteriology. The description of the novelties here are well and abundantly illustrated. Many—we might say most—of the paragraphs here inserted seem to have been gleaned from the *Chemiker Zeitung* and its Supplement the *Chemisches Repertorium*.

We hope that the "Nouveautés Chimiques" will be continued yearly, as it is calculated to prove highly serviceable to men of Science.

A Manual of Chemistry, Theoretical and Practical. Based on Watts's Edition of "Fownes's Manual." By W. A. TILDEN, D.Sc., F.R.S., Professor of Chemistry in the Royal College of Science. London: J. and A. Churchill. 1897. Crown 8vo., pp. 599.

THIS work is somewhat complicated in its origin. The last traces of the work of Fownes, we are told, have disappeared under the hands of the successive editors in accordance with the development of chemical science, and the final result is a most satisfactory specimen of the intermediate type of chemical handbook.

A preliminary chapter has been added, giving in broad outline the leading points in the history of chemistry from the time of Boyle downwards. The author expresses

the very correct opinion that such historical survey will prove useful to the student.

Dr. Tilden, whilst accepting the periodic system, does not regard it as the be-all and end-all of classification. It is pointed out as a singular fact that the discoverers of oxygen, Scheele and Priestley, remained phlogistians to the end of their lives.

After the list of the elements we find the term "metalloids" used, not as it is done in France, for the non-metals, but as a sub-class of the latter.

In speaking of the composition of the atmosphere, we find an expression which may be misunderstood. The writer means, of course, that ozone and hydrogen peroxide may replace each other, but a junior reader might carry away the impression that ozone is a synonym for hydrogen peroxide.

Dr. Tilden greatly underrates the confusion arising from the different hydrometer scales, which are far more than two or three.

The periodic law is here ascribed to Newlands, Lothar Meyer and Mendeleeff receiving the credit of having elaborated the original idea. The relations among the atomic weights of the elements, as connected with their attributes, are explained by the diagram devised by Dr. Emerson Reynolds, and subsequently modified by Mr. Crookes. Dr. Tilden does not enter upon a variety of other schemes for the classification of the metals, such as those of Chancourtois.

The section on the glass manufacture gives the composition of Bohemian plate-glass and English flint-glass, but overlooks the Jena glass, which is now rapidly and deservedly rising in favour.

According to the recent researches of Prof. Berthelot, pure copper (commercially so called) was used for tools and weapons before the introduction of bronze.

In speaking of the Bessemer process, it might have been added that the slag from the basic process (Thomas slag) is an excellent phosphatic manure, equal—and in some cases even preferable—to superphosphate.

Tin has occasioned some trouble in chemical classification. In common life it ranks as a metal, and is, indeed, one of the bodies upon which the concept of a "metal" was primarily founded. We sometimes, however, find it placed as a non-metal or a semi-metal. Dr. Tilden associates it with thorium, zirconium, and cerium.

It is impossible for us to notice all the numerous passages in this work which justify, and indeed call for, favourable comment. The index is excellent.

The Organised Science Series. First Stage.—Inorganic Chemistry. By G. H. BAILY, D.Sc. (Lond.), Ph.D. (Heidelberg), Lecturer in the Victoria University, and also Assistant Examiner in the Science and Art Department. Edited by WILLIAM BRIGGS, M.A., F.C.S., F.R.A.S., Principal of University Correspondence College. London: W. B. Clive, University Correspondence College Press. Warehouse, 13, Booksellers' Row, Strand, W.C. 8vo., pp. 210.

THE title-page of this little book might justify a few questions. What, for instance, is "Organised Science"? The definition of Science is "organised knowledge"; what, then, is "organised science"? Or is it the series only which is organised, rather than any other series? Again, it may be asked, what are the characteristic features of University Correspondence College, an organisation which seems to do its own printing and publishing? Again, we find mention of a "University College Tutorial Series," which has the same general editor. How are two series connected together, if at all? There is further a weekly journal, the *University Correspondent* and *University Correspondence College Magazine*. There is, if it has not experienced the "happy despatch," a journal bearing the ominous name, the *Competition*, but issued, as far as we are aware, under different auspices. Have

we not here the old unhappy game of too many cooks spoiling the broth?

It will be perceived that the "University Correspondence College" prepares students for examinations at the London University. The various chapters are fitted with sets of questions, and, in addition, there are answers to the questions and to the "chemical calculations."

The instructions given are satisfactory, but the question still arises *Cui bono*?

Abstract of Chemical Analysis. Second Part.—Quantitative Analysis. By E. FINX, Head of Practical Operations in Analysis at the Municipal School of Industrial Physics and Chemistry of the City of Paris. ("Precis d'Analyse Chimique." Deuxième Partie. — Analyse Quantitative. Par E. FINX, Chef des Travaux Pratiques d'Analyse à l'Ecole Municipale de Physique et de Chimie Industrielles à la Ville de Paris). Paris: Georges Carré and C. Naud. 1896.

THIS book differs little from the generality of works on the same subject and of the same extent. The balance proposed by Dr. Curie is described and figured. The weights are of course arranged on the ultra-decimal principle, with the exclusion of all pieces which are not sub-multiples of the preceding weight. The bases are classified as belonging to the group of arsenic, copper, iron, barium, and potassium.

The analytical methods are divided into the gravimetric, electrolytic, volumetric, and colorimetric. The instruments employed in the last method are those of Duboscq and Salleron. The tintometer is not mentioned. Quantitative spectroscopic analysis also is overlooked.

Practical Work in Physics. For Use in Schools and Colleges. By W. G. WOOLCOMBE, M.A. (Oxon), B.Sc. (London), Senior Science Master in King Edward's High School, Birmingham. Part III. — *Light and Sound.* Oxford: Clarendon Press. London: Frowde (Oxford University Press Warehouse), Amen Corner, E.C. Crown 8vo., pp. 95.

THE Preface informs us that an essential feature of the work before us is to furnish, at a trifling cost, a fairly complete experimental course in the subjects covered. As regards the trifling cost we have to consider, in the first place, the book itself; and in the second, the inexpensive character of the apparatus required. The subjects here considered "do not appeal so much to the student's power of observation as to that of his judgment." Here therefore the author, in his estimate of the educational value of physics, differs from those authorities who consider that its value consists mainly in its training and stimulating the student's faculties of observation. Who is in the right we do not undertake to decide. It will be perceived that sound is not on all fours with light, heat, and electricity. These agencies are all *molecular*, whilst sound is plainly *molar*. So that, in spite of the fact that the study of sound first made us familiar with vibratory motions, we are at a loss as to whether acoustics ought to rank with optics.

Quantitative Estimation of Urine; New System of Rapid Analysis, for Medical Men and Pharmacists. By J. BARKER SMITH, L.R.C.P. (Lond.). London: Baillière, Tindall, and Cox. 1897. Pp. 37.

THE author of this pamphlet has had a long experience in the analysis of urine, and has naturally evolved, so to say, several new and rapid methods. Time is, of course, a great factor in carrying on work of this character, and the general practitioner will doubtless appreciate the improvements and modifications herein described.

The tests applied to urine may be arranged in two main divisions:—

- I. Tests of a sample of urine rendered *alkaline*, these being used for the estimation of *acidity, urea, sugar, total urates, phosphates, ammonia, &c.*
- II. Tests associated with the estimation of normal or acid urine:—*albumen, biliary salts, peptone, &c.*

The general procedure (a) is fully set forth and explained; this is followed by two sections (b and c), fully describing the special methods used, all the details being minutely gone into; possible errors and misinterpretations of results are pointed out, and many examples of actual periodic examinations are given.

Accompanying this pamphlet is a small, conveniently arranged pocket set of tables, and general instructions for the rapid thermometric method of the quantitative analysis of urine; the complete apparatus required (besides these tables) comprises only a measure, phial, and thermometer, so that with the few reagents required only a small box is needed.

CORRESPONDENCE.

THE NEW SCIENTIFIC CLUB.

To the Editor of the Chemical News.

SIR,—My attention has been drawn to the fact that my name appears on a circular signed by Mr. Robert Ingram proposing to found a Club for scientific men. I write to ask you to give publicity to the fact that I know nothing of the proposed Club, and have not sanctioned the use of my name in any way.—I am, &c.,

W. RAMSAY.

12, Arundel Gardens, W.,
April 9, 1897.

CHEMICAL SOCIETY ELECTION.

To the Editor of the Chemical News.

SIR,—Your readers might be led to infer from Professor Ramsay's letter of April 3rd that I had not replied to the communication he addressed to me: this would be a mistake.

Professor Ramsay well knows that I made a full reply, in which I challenged him to put aside all verbal quibbles; and that it is I who am without an answer. He has but to say that he wishes that the letter he wrote to me, dated 27th March, and my answer to it, dated 30th March, shall be published.—I will then forthwith place both in your hands for communication to your readers.—I am, &c.,

HENRY E. ARMSTRONG.

TEACHING OF CHEMISTRY.

To the Editor of the Chemical News.

SIR,—I have read the letters of Mr. Woodward and Mr. Wigley with great interest, but cannot see in either sufficient reason to change my original position. Both the above-named gentlemen think I over-estimate the danger beginners incur when handling the simple gases. I can only say that I know of a number of serious explosions of these gases that I believe would have been avoided had their preparation been deferred till later in the course. Of course I cannot speak of English schools, but in the American schools with which I am familiar this danger is greatly increased by giving too many pupils to one teacher. In my opinion this overcrowding of classes is the most serious shortcoming in our American schools to-day, but it only aggravates the danger which is already present. In regard to the danger of explosions from careless handling of acids that is present in either quali-

tative analysis or the preparation of the non-metallic elements, and so cannot count against either subject, Mr. Wigley thinks qualitative analysis would not prepare the student to work with gases. I think work with chemicals and apparatus in any branch of the subject will materially aid work in other lines.

Both Mr. Woodward and Mr. Wigley seem to place small value on the educational effect of qualitative analysis. To my mind one of the most important objects of laboratory work is to teach the student to think logically and to observe accurately. Both of these objects are as well accomplished by qualitative analysis as by the preparation of the simple gases. Whether qualitative analysis becomes routine or not depends entirely upon the ability of the teacher. Surely a subject that has claimed the attention of some of the ablest chemists who have ever lived ought to contain enough intellectual matter to prevent the subject from becoming stale and profitless to the beginner. With the rare elements to study—which I should not advise at this stage of the work—I think there is no need for purely mechanical work.

To Mr. Woodward's criticism that the students *won't* reason, I may say I think we are all more or less lazy in that respect. The remedy I suggest is the one Nature uses for more advanced scholars, namely, to stimulate interest by withholding results from the experimenter. I strongly approve of text-books in which the students are never given a result when it can reasonably be withheld.

I may state my position by saying that I do not advise the abandonment of work with the simple gases and the other non-metallic elements, but I suggest that it is not suitable for the first work in the chemical laboratory. I should recommend some such chemical course as the following:—Recitations, with strict and searching questioning daily, upon the non-metallic elements and such work as is usually included in that branch of the subject in a short text-book. These recitations should be thoroughly illustrated by experiments performed by the teacher, which are recorded by the pupils. Then the essential principles of qualitative analysis should be taught by laboratory work. Finally, a review of the non-metallic elements, in which the student does the actual work himself. Here quantitative analysis could be brought in to great advantage, I think. I am not prepared to say that it ought not to be brought in sooner, for I think it is too little used; but I *think* some knowledge of chemical manipulation had best precede it. Qualitative analysis can be made quantitative also with great ease. After this review of the non-metallic elements, the subject is ready to be expanded along any line the teacher may see fit to follow.

The delay in answering the letters of Mr. Woodward and of Mr. Wigley, and the fact that I answer both gentlemen in one and the same letter, I hope will be pardoned on account of my distance from London.—I am, &c.,

ALFRED C. BEEBE.

Savanna, Ills., U.S.A.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 13, March 29, 1897.

The President announced the presence of Dr. Nansen at the session, whom he cordially welcomed on behalf of the Academy.

Transformations of the Sugars, and on Levulic Acid.—MM. Berthelot and André.—In virtue of its gene-

ration by means of aldehydic groups, the molecule of glucose behaves as being eminently moveable, capable of being split up at the ordinary temperature in various different directions: its constituent atoms of hydrogen and oxygen oscillate among numerous centres of carbon. In all cases it is a question of purely chemical agents. We have never ceased to think that it must be the same in the production of alcohol; the living cell which there intervenes not being the true specific agent of the reaction, but having the function of secreting such a specific agent. To this opinion adhere the majority of the physiologists who occupy themselves with the study of infectious maladies.

On the Fatty Matters Found in the Egyptian Tombs of Abydos.—C. Friedel.—The author has examined certain antique objects found at Abydos by M. Amélineau and considered to be anterior to the first dynasty. The fatty matter consisted chiefly of palmitic and stearic acids, and was doubtless the tallow of beef or of mutton. It is interesting to find that the fatty acids, such as the stearic and palmitic acid, and even the glycerides of these acids, have been capable of preservation for thousands of years. Among the substances found in small vases was pulverised lead sulphide mixed with a quantity of fatter matter; evidently a cosmetic used as antimony sulphide is still employed in the East.

Transformation of the Diamond into Graphite in the Crookes Tube.—H. Moissan.—Mr. Crookes has demonstrated in his fine researches on the phenomenon which he has named molecular bombardment that on placing diamonds in one of his tubes they quickly lose their lustre and are coated with a black layer. Having been present in his laboratory at this curious experiment I asked him for some of the diamonds which had been thus bombarded that I might study the variety of carbon produced under these conditions. Mr. Crookes having kindly sent me a diamond the surface of which had been completely blackened by this bombardment, I heated it to 60° in an oxidising mixture of potassium chlorate and fuming nitric acid prepared from sulphuric acid exactly monohydrated and potassium nitrate fused and quite free from moisture. The action on the black layer is very slow. There is produced graphitic oxide, which at an increased temperature yields pyrographitic acid which is easily destroyed by nitric acid. Hence the variety of carbon which coated the diamond was graphite. This transformation of the diamond into graphite must be very high. Mr. Crookes had already proved the platinum-iridium can be fused in his tubes, but the temperature obtained in the bombardment is much higher, since the transformation of diamond into graphite requires the high temperature of the electric arc. The higher the temperature to which graphite is raised the greater is its resistance to oxidation. The temperature reached is probably about 3600°.

Mutual Actions of Electrodes and of Cathodic Rays in Rarefied Gases.—H. Deslandres.—The author concludes that when we have in the vicinity of a cathode a conductive or insulated body which is taken as an anode or is insulated, everything ensues as if the cathodic rays were attracted. The mutual action of the rays and the cathodes takes place only when the rays interpenetrate each other.

Stannic Chlorobromides.—A. Besson.—Theory leads us to foresee the existence of the chlorobromides of the type SnX_4 , SnCl_3Br , SnCl_2Br_2 , SnClBr_3 . The compound SnCl_3Br forms the main part of the fraction which distils at 50–55° under the pressure of 3 c.m. The chlorobromide, SnCl_2Br_2 , is separated from the fraction passing over between 60° and 70°. The compound SnClBr_3 distils over the same pressure at about 73°.

Conditions of the Direct Combination of Sulphur and Hydrogen.—H. Pélabon.—Hydrogen may still combine with sulphur as long as the temperature is not below 215°. Between 215° and 350° the combination is still

limited. The direct combination of the two substances is effected the more rapidly the higher is the temperature. The maximum quantity of hydrogen sulphide formed at a given temperature increases regularly with temperature. Above 440° we reach the same limit, whether we set out from sulphur and hydrogen or from pure hydrogen sulphides. If we substitute for pure hydrogen a mixture of hydrogen and nitrogen, the maximum quantity of hydrogen sulphide formed after heating for a given time is less than with pure hydrogen; all circumstances being equal the difference is less the higher the temperature.

Action of Bromine and Hydrobromic Acid upon Ethyl Acetate.—Boleslas Epstein.—A critique of the results of M. Crafts published in the *Comptes Rendus*, vol. lvi., p. 707, 1863.

MISCELLANEOUS.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on April 5th, Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, presiding. The following were elected Members:—Mr. J. H. Colls, Mr. H. E. Diamond, Mrs. J. Dundas Grant, Mr. Douglas Hall, Mr. W. Hunter, and Mr. F. M. Mackenzie, M.R.C.S. The special thanks of the Members were returned to Sir William J. Farrer for a donation of £50 to the Fund for the Promotion of Experimental Research at Low Temperatures.

Gravimetric Estimation of Sugar.—G. Ambuhl (*Chem. Zeit.*, 1897, xxi., 137).—The author recommends that the cuprous oxide produced in the Fehling-Alilihn process should be dried for one hour at 98.5° C., and weighed as such, instead of being reduced to the metallic state. He presents an elaborate table, showing the results of the method when applied to forty-six samples of wine, honey, and diabetic urine. In the case of wines, the figures are practically identical with those obtained by weighing the metal, being usually a trifle higher (maximum +0.09; average +0.034 per cent). With honey, the excess averages 0.23 per cent on amounts of sugar varying from 57 to 69 per cent; and with urine containing 4.25 to 6.16 per cent of sugar, the mean difference between the two processes is 0.10 per cent, but in this instance the suboxide was manifestly contaminated with organic substances, which suffer decomposition on ignition.—*The Analyst*.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Gun Paper.—Will some correspondent inform me the way to make gun paper, by the quire at a time. I find when trying to make more than a single sheet the whole quire adheres and becomes one solid cake or block.—F. F.

Pemberton's Molybdate Method for Phosphoric Acid.—Will some correspondent kindly inform me where I can find any description of a modification of Pemberton's molybdate method for phosphoric acid, being the use of glue to make the yellow precipitate sink so as to enable one to better see the end reaction. The method is used, I believe, in the State Agricultural Laboratories in Switzerland.—R. C. W.

ERRATA.—No. 1910, p. 11, col. 1, line 19 from bottom, for "0.9°" read "0°". No. 1950, p. 179, col. 2, line 5 from bottom, for "Staining" read "Storing."

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1952.

METHOD OF SEPARATION OF NICKEL FROM COBALT, NICKEL FROM IRON, AND COBALT FROM ALUMINIUM.

By E. PINERUA,
Laboratory of the University of Valladolid, Spain.

ON account of the very close resemblance between nickel and cobalt their separation offers many difficulties, and calls for special methods. The process generally employed, viz., the potassic nitrite method, is very tedious, and above all requires certain conditions as to the proportion of cobalt present (Fleitmann, *Z. fur. Anal. Chem.*, xiv., 76, 1875).

Liebig's method, by means of cyanide of potassium, bromine, and an alkali, is very delicate.

That of Langier, which consists in precipitating the nickel and cobalt as oxalates, and dissolving these in ammonia, and exposing this solution to the action of the air, has been abandoned.

Gautre's modification (*Z. fur Anal. Chem.*, v., 75, 1866) of the cyanide of potassium and mercuric oxide process, is again not very exact.

The latest method, that of Knorre and Ilinski (*Z. fur. Ange. Chem.*, vi., 264, 1893), based on the precipitation of cobalt by nitroso-β-naphthol, which does not precipitate the nickel, is good; but we think the simplest and most rapid is that which we have recently employed at this laboratory, founded on the insolubility of chloride of nickel in a solution of ordinary ether saturated with hydrochloric acid gas at a low temperature.

The action of hydrochloric acid gas on the metallic oxides and their salts has been studied by Debray, E. Pechard, Ditté, Engel, Hanriot, Rothe, Smith and Oberholtzer, Smith and Hibbs, Smith and Meyer, Jannasch and Schmith, Bird Moyer, F. A. Gooch, F. S. Havens, and many other chemists; but we believe that, up to the present, no method of separation of the above-named metals has been published, based, as ours is, on the insolubility of the chlorides of nickel and aluminium, and the great solubility of cobalt and iron, in the solution previously mentioned.

The hydrated chlorides (0.3 grm. to 0.4 grm.) of the metals, nickel and cobalt, nickel and iron, cobalt and aluminium, are dissolved in the *smallest possible quantity of water*, and to the solution is added 10 or 12 c.m. of fuming hydrochloric acid and 10 c.m. of ordinary ether (D 15 c.=0.725), or, better still, anhydrous ether. This must be well shaken, and through the resulting homogeneous liquid a current of hydrochloric acid gas is passed continuously until complete saturation, at the temperature of melting ice, with which the beaker or test-tube should be surrounded.* During this operation the nickel is precipitated in the state of heavy, crystalline, *yellow chloride*, and the cobalt remains in solution with an intense blue colour (Engel's acid chloride). In the presence of iron, which under these conditions would give a bright yellow solution, the colour becomes green.

* Our hydrochloric acid gas was obtained by the reaction in the cold of concentrated sulphuric acid and chloride of ammonium. The apparatus employed consists of a litre flask, with three large openings at the top, and one draw-off tube at the side near the bottom. One of the three necks allows the gas to pass by means of stoppered tube; another serves for the introduction of small lumps of the ammoniacal salt; and in the third is inserted a *Welter* safety tube, by which means small quantities of sulphuric acid are added. The lower side tube is used for emptying the apparatus when necessary.

Most of the commercial chlorides of nickel and cobalt, *soi-disant* pure, are really very impure. By using our process for precipitating the nickel, on the commercial chlorides of cobalt, the resultant liquid is of a bluish green instead of a pure blue colour, indicating the presence of iron.

The precipitated chloride of nickel is yellow, but sometimes the remaining liquid is of a light yellowish green colour, indicating the presence of iron and cobalt.

We have noticed, by using different solvents, phenomena which by their importance demand studying with very great attention. We are led to believe, as are the chemists Krüss and Schmidt, Remler, Winckler, de Koninck, and others, that nickel and cobalt are not actually known in a state of purity, and that they probably contain other elements still unknown.

The precipitate of chloride of nickel is washed by decantation, with ether saturated with hydrochloric acid gas, at a low temperature, collected on a filter, and thoroughly washed again on the filter; it can then be weighed and the nickel estimated in the ordinary manner (such as sulphate, for example) with very accurate results.

The same method of procedure can be utilised for the separation of aluminium and cobalt; the former metal is completely precipitated in the state of insoluble chloride, as it is when in the presence of iron, using the analogous method of Hanriot and Rothe, as modified by F. A. Gooch and F. S. Havens.*

The cobalt remains like the iron in solution; and the insoluble chloride of aluminium, washed with ether saturated with hydrochloric acid gas, can be estimated with great accuracy.

To effect the separation of nickel from iron, it is necessary to wash the chloride of nickel many times with ether to eliminate the chloride of iron which is re-dissolved, and the operation must be repeated several times.

THE OCCURRENCE OF RAFFINOSE IN AMERICAN SUGAR BEETS.

By W. E. STONE and W. H. BAIRD.

RAFFINOSE, as a distinct kind of sugar, belongs to the comparatively little met with class of tri-saccharides with the formula $C_{18}H_{32}O_{16}$. It has been shown that the sugars found in different plants, such as Eucalyptus, cotton-seed, barley, wheat, and finally in molasses and refinery products of the sugar beet, and called variously melitose, gossypose, and raffinose, are all identical with each other.

In the residual and secondary products resulting in the manufacture of beet sugar, raffinose has long caused serious and unexplainable errors, inasmuch as it crystallises with cane sugar, modifying its form and increasing the specific rotation. It was at first thought that raffinose was not originally present in the beet juices, but it has been since shown that such is not the case.

The special processes of making beet sugar from beet juices, as carried on at the Norfolk (U.S.A.) Works, are fully described; an important point to note is that, owing to the behaviour of raffinose during the process of manufacture of beet sugar, it gradually becomes more and more concentrated in the molasses and secondary products of the factory, and it has been noted that if the amount of raffinose present reaches 8 to 12 per cent, or even less, it not only destroys the accuracy of all polarimetric determinations, but seriously retards the crystallisation of the sucrose itself.

* "Method for the Separation of Aluminium from Iron," by F. A. Gooch and F. S. Havens. Contributions from the Kent Chemical Laboratory of Yale University. *American Journal of Science*, vol. ii., Fourth Series, December, 1896; *CHEMICAL NEWS*, vol. lxxiv., p. 296.

Repeated attempts, during 1895 and 1896, were made to detect raffinose by Tollens's method of producing mucic acid, but were met with failure, and the results herein given well illustrate the untrustworthiness of the mucic acid test when applied to complex mixtures of salts and organic matters, such as molasses. Numerous methods have been proposed for the isolation of raffinose, and the authors proceed to describe several of them, such as those used and described by Kodyl and Scheibler, and they finally found a combination of several of these which proved successful. This method is described at length, and from the results obtained the authors feel justified in concluding that raffinose occurs in the juices of the American sugar beet in appreciable quantities. Certain peculiarities of crystallisation of mixtures of sucrose and raffinose are pointed out, the authors finding, when examining the crystals under the microscope, that frequently solutions containing apparently but a very small amount of raffinose would completely crystallise into forms which could scarcely be distinguished from pure raffinose. This modification of the crystalline form of sucrose would seem to afford a ready means of detecting the presence of small amounts of raffinose when mixed with sucrose.—Abridged from the *Journal of the American Chemical Society*, xix., No. 2.

THE DETERMINATION OF SULPHUR IN CAST-IRON.

By FRANCIS C. PHILLIPS.

IN a Paper read before the American Chemical Society in August, 1895 (*Journ. Amer. Chem. Soc.*, xvii., 891), I have detailed some experiments made in the determination of sulphur in white cast-iron by the evolution method, and have attempted to show that the loss of sulphur in its determination in such iron may be due to the formation of organic sulphur compounds not oxidisable to sulphuric acid by the usual means.

By passing the gases evolved during the solution of the iron in hydrochloric acid through a heated porcelain tube, it was found that the volatile organic sulphur compounds may be decomposed and nearly all the sulphur recovered by conversion into hydrogen sulphide, oxidation and precipitation as barium sulphate.

In judging of the correctness of an analytical method it has been necessary, in the case of the majority of the constituents of iron, to depend upon a single criterion; that method is regarded as most accurate which, being correct in its details, yields the highest percentage of the constituent sought to be determined. For it is hardly possible to add to pure iron a known percentage of sulphur, phosphorus, or carbon, and test the method by a determination of the added constituent. For the determination of sulphur in iron it has been common to regard the method of oxidation and solution of the iron by nitric acid, followed by precipitation of the sulphur in form of barium sulphate, as the most accurate, inasmuch that it yields results somewhat higher than those obtained by other modes of procedure.

It does not seem probable that an appreciable error could occur in the use of this method, unless, in the simultaneous oxidation of the carbon and sulphur of the iron, an organic sulphur compound should be formed.

It has seemed to be of interest, however, to apply a method for the determination of sulphur by which all the constituents of the metal could be completely oxidised in a dry state and at a high temperature, in order to avoid as effectually as possible the chances of loss due to the conversion of sulphur into a volatile compound not oxidisable by ordinary means to sulphuric acid.

In searching for a method which should answer these requirements, it seemed possible that by heating the iron in the form of fine powder, in presence of a mixture of alkaline carbonate and nitrate, the sulphur might be

oxidised directly and completely to the condition of a sulphate without affording an opportunity for the escape of a trace of sulphur in some intermediate volatile or soluble compound. Accordingly an experiment was tried in the following way:—

An iron containing its carbon in the combined form was melted in a crucible, and poured while fused into water. The granulated metal was crushed in a steel mortar to an extremely fine powder. The powder so obtained was sifted through bolting sheeting.

Two and one-half grms. of the sifted iron were mixed with 10 grms. of a mixture of equal parts of sodium nitrate and carbonate in a platinum crucible. The crucible was covered and heated over a Bunsen burner. At a red heat a sudden and rather violent reaction occurred, and, having been begun, was easily maintained with very little aid from the burner flame. The reaction appeared to be complete in a few minutes. After heating for a half hour the crucible was cooled, and its contents softened in water. A residue of a reddish brown powder, consisting of ferric oxide with a little ferrous oxide, was obtained. This residue was found to contain no sulphuric acid, and, on digesting with hydrochloric acid, dissolved without effervescence, showing that none of the particles of the original iron had remained unoxidised. From the results of this experiment, and others which need not be detailed here, it seemed to be possible to oxidise finely divided iron so completely by heating with sodium carbonate and nitrate, that its sulphur might be converted quantitatively into sulphuric acid.

The mixture of sodium carbonate and nitrate, although tending to oxidise finely divided iron, seems to exert a less powerful action upon the carbon contained in the iron, and this carbon may appear as a black residue after the fused mass has been softened and extracted by water and the ferric oxide dissolved in hydrochloric acid.

It seems to be important for the success of the method that in the oxidation of the iron the carbon should also be nearly or completely oxidised, for if the carbon remained unburned a portion of the sulphur might escape oxidation. In general it may be said that the order of oxidation of these three elements by the method used is as follows:—1, iron; 2, carbon; 3, sulphur; the iron being the most easily oxidised, and the sulphur the most difficult to oxidise. This order is not exactly what we should anticipate; but it is to be remembered that unless the iron grains are fine enough to be penetrated by oxygen, and changed completely into a soft powder of ferric oxide, the sulphur and carbon have no opportunity to oxidise at all. If the iron could be used as an impalpable powder the order of oxidation would probably be different. The marked resistance of the carbon to oxidation has been frequently observed, even when using more sodium nitrate in the fusion than is theoretically enough to completely oxidise both iron and carbon, supposing that the sodium nitrate is reduced only to nitrite in the process.

Experiments of a similar kind were tried with ferromanganese. A metal containing about 80 per cent of manganese was used. By crushing in a steel mortar this iron was very easily reduced to a powder fine enough to pass through bolting sheeting. On heating the powder with the mixture of sodium nitrate and carbonate a most violent reaction occurred, the metal burning with a long flame, extending several inches above the crucible. In order to control the reaction it was found necessary to melt one-half of the fusion mixture to be used in the crucible, and then add slowly the other half, previously mixed with the powdered metal, while stirring constantly. In this way the reaction could be easily controlled. On softening the fused mass in water it was found that the iron had been peroxidised and the manganese changed to binoxide. No trace of sodium manganate was ever formed, the solution in water being after filtration invariably colourless. No carbon was found in the residue. The oxidation of the carbon is much more easily effected

in the case of iron containing a high percentage of manganese. In all the trials made the silicon of the iron was oxidised, but it was found that when the fused mass is softened in water very little silica enters into solution as an alkaline silicate, the greater portion remaining insoluble and in a flocculent form.

Experiments were then tried with a grey iron. This form of iron could not be crushed to a fine powder, and an experiment was made in reducing it from small drillings by means of a chilled iron rubber and plate, such as is ordinarily used for grinding ores. Several grey irons were tried in this way. Some could not be powdered by the method just mentioned, the grains tending to flatten instead of being crushed. Others were readily reduced, but the powder was not in any case fine enough for sifting through bolting sheeting. It was found, in the case of a grey iron reduced to powder by the method of grinding, that on fusion with the mixture of sodium nitrate and carbonate, used in the preceding experiments, the graphitic carbon of this iron was more readily burnt than the combined carbon of white iron.

As it had proved to be a somewhat difficult matter to oxidise completely the carbon of the iron in the various experiments made with the fusion method, notably in the case of white iron, some trials were made in the use of sodium peroxide. This proved to be a more efficient oxidising agent for iron and its contained carbon than sodium nitrate. For these trials a mixture was used consisting of forty-five parts each of sodium peroxide and sodium nitrate, together with ten parts of sodium carbonate.

White iron was oxidised and its carbon burnt during a fusion lasting less than ten minutes.

On heating ferromanganese with this mixture the iron was found to be completely oxidised. The carbon was burnt, and the manganese was oxidised and converted into sodium manganate, yielding a deep green solution when the fused mass was digested in water.

An admixture of sodium carbonate to sodium peroxide tends in all cases to diminish its action upon finely-divided iron at a high temperature, and renders the process more easily controlled. It seemed to be possible to base a method for the quantitative determination of sulphur in certain kinds of cast iron upon the reactions described above.

An indispensable condition of success in the use of the method is found in the extreme fineness of the iron. In the case of white irons the fineness of the powder has been secured by crushing in a steel mortar until the powder passed through a sieve of bolting sheeting or bolting cloth.*

Some grey irons cannot be crushed or ground. To these the method is not applicable. For grey irons, however, the evolution method answers all requirements.

The following details are given of the method finally employed:—

1. *White Iron*.—About 1½ grms. of the finely-powdered and sifted metal was intimately mixed with 8 grms. of the sodium peroxide mixture above mentioned, or with 4 grms. each of sodium carbonate and nitrate. The somewhat violent reaction set up on the application of strong heat to the platinum crucible was completed in a few minutes. The crucible was heated for about twenty minutes in all. After cooling, the contents were softened in water, the solution decanted, and the residue ground, while wet, in a mortar. The solution and residue were then digested in a beaker on the water-bath for one hour after addition of 2 c.c. of strong bromine water. The liquid was then

Character of iron used.	Fusion mixture employed.	P.c. of sulphur found by fusion.	P.c. of sulphur found by the method of oxidation by nitric acid.
White iron A crushed in mortar and sifted through bolting sheeting.	Contained equal parts of sodium carbonate and nitrate.	0'112	0'101
		0'112	0'098
		0'111	0'096
		0'107	0'099
		0'114	0'100
		0'114	0'102
		0'106	0'102
		0'108	0'104
		0'107	
		0'103	
Means	0'109	0'100	
White iron B crushed and sifted.	Contained— 45 parts NaNO_3 45 parts Na_2O_3 10 parts Na_2CO_3 .	0'155	0'143
		0'150	0'149
		0'130	0'143
		0'139	0'147
		0'166	
		0'156	
		0'156	
		0'161	
0'151			
Means	0'151	0'145	
Ferromanganese crushed and sifted.	Contained equal parts of sodium nitrate and carbonate.	0'022	0'012
		0'027	0'013
		0'018	0'012
		0'018	0'010
		0'018	
		0'019	
		0'016	
Means	0'020	0'012	
Grey iron drillings powdered by rubber and plate. Not sifted.	Contained equal parts of sodium nitrate and carbonate.	0'034	0'027
		0'030	0'030
		0'036	0'026
		0'034	0'028
		0'033	0'028
		0'034	0'022
Means	0'033	0'027	

filtered, acidulated with hydrochloric acid, evaporated to dryness to separate the small portion of silica which had entered in solution, and filtered. The sulphuric acid was determined in the filtrate in the usual manner. The barium sulphate obtained was always white. If the fusion mixture contains sodium carbonate and nitrate, but no sodium peroxide, the crucible must be heated for a longer time, but a portion of the carbon of the iron may still remain unoxidised.

2. *Ferromanganese*.—In this case it is better to use a mixture of equal parts of sodium nitrate and carbonate, omitting the sodium peroxide.

Ten grms. of the mixture were divided into two portions, one of which was fused in a crucible. The other portion, mixed with 2 or 2½ grms. of the finely-powdered iron, was then slowly added. Although too violent combustion of the iron is to be avoided, it seems to be important, for the success of the method, that a reaction of decided intensity should occur during the fusion.

Sodium nitrate possesses an advantage over sodium peroxide in its greater purity, the former compound being readily obtainable with practically insignificant traces of sulphur.

Natural gas was the fuel used for the Bunsen burner in heating the charges. This gas was found, by repeated experiments, not to contain a sufficient quantity of sulphur

* Two different materials are sold which are suitable for the sifting. One is called bolting cloth, the other bolting sheeting. The bolting cloth used in these experiments contained about eighty-five meshes to the linear inch, while in the bolting sheeting about one hundred and thirty-five were counted. The material having the smaller number of meshes is made of coarser threads, however, and yields, on account of the smaller openings, a finer powder. Bolting cloth is, on this account, better suited to the preparation of a sample of white iron for a determination of sulphur by the method described.

to affect the purity of the sodium carbonate when heated in a platinum crucible in the same manner as in the case of the determinations described.

The usual occurrence of sulphur compounds in coal-gas would preclude its use in the application of the method.

From the experiments, the results of which are stated in the accompanying table, there seems to be some reason to suppose that not quite all the sulphur of the iron is converted into barium sulphate when the metal is oxidised and dissolved by nitric acid. That it has been completely recovered by the process of fusion cannot be positively asserted.*

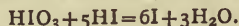
The method I have described is not proposed as a substitute for any existing method. The purpose of the present work was merely to ascertain as far as possible whether, by a process of direct oxidation of the iron in a dry state, a larger proportion of the sulphur could be recovered in weighable form than by the usual method of oxidation and solution in nitric acid.

My thanks are especially due to Mr. F. B. Smith for great care and attention to detail in conducting the experiments I have detailed.—*Journal of the American Chemical Society*, xviii., 1079.

THE APPLICATION OF IODIC ACID TO THE ANALYSIS OF IODIDES.†

By F. A. GOOCH and C. F. WALKER.

It has long been understood that iodic acid is easily and completely reduced by an excess of hydriodic acid with the liberation of iodine according to the equation—



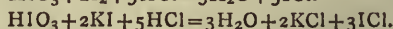
To apply this reaction to the quantitative estimation of iodic acid, it is only necessary to add to the free iodic acid or soluble iodate an excess of a soluble iodide, to acidify—best with dilute sulphuric acid,—and to titrate the iodine thus set free with sodium thiosulphate, one-sixth of the iodine found being credited to the iodic acid.

It has been shown recently by Riegler (*Zeit. Anal. Chemie*, xxxv., 305) that this reaction may be also applied to the quantitative estimation of iodides, the iodine set free upon the addition of a known excess of iodic acid to the iodide solution being removed by petroleum ether, and the residual iodic acid titrated directly with sodium thiosulphate.

The present investigation was undertaken to define more particularly the limit of applicability of the reaction and to establish, if possible, a direct method for the quantitative estimation of iodides, dependent upon the action of iodic acid or an iodate in the presence of free sulphuric acid, neutralisation of the solution by means of an acid carbonate, and titration of the free iodine by arsenious acid—five-sixths of the iodine thus found being credited to the iodide to be estimated. It has been found that by fulfilling certain necessary conditions, the proposed method is entirely successful, so far as concerns the estimation of iodine in iodide solutions free from large amounts of chlorides or bromides.

In a system containing a considerable quantity of free iodine with variable amounts of the other reagents mentioned, as well as possible impurities, it is conceivable that secondary reactions may occur, depending largely on conditions of mass, time, and temperature, and of a sort likely to alter the amount of recoverable iodine, or to

exert an excessive oxidising influence on the arsenious acid finally titrated. It has been established by Schönbein, Lunge and Schoch, and others, that iodine forms compounds with the alkalis of the type $\text{R}-\text{O}-\text{I}$, and Phelps (*Am. Journ. Sci.*, ii., 70, 1896) has recently found that the formation of some such compound, accompanying the iodate naturally expected, is distinctly recognisable when iodine and barium hydroxide interact at ordinary temperatures. It has been shown, also, in a former paper from this laboratory (Roberts, *Am. Journ. Sci.*, xviii., 157) that free iodine or an iodide interacts very easily with iodic acid in the presence of dilute hydrochloric acid with the formation of iodine monochloride, according to the equations—



Moreover, organic compounds containing the groups $-\text{I}=\text{O}$ and $-\text{I}=\overset{\text{O}}{\underset{\text{O}}{\text{O}}}$, in which iodine seems to be analogous to nitrogen, result in great variety from the oxidation of halogen substitution products. It would seem, therefore, that the formation of inorganic reduction products of iodic acid under the conditions likely to obtain in this analytical process might be by no means beyond the bounds of possibility.

A few simple qualitative tests to determine the possibility of interaction between small quantities of iodine and iodic acid alone met with negative results. Thus, a single drop of a decinormal solution of iodine, made as usual in potassium iodide, gave when added to 10 c.m.³ of decinormal iodic acid a distinctive colour to chloroform. Similar results were obtained when the iodine was employed in aqueous solution in which there was no alkaline iodide. A few drops of an aqueous solution of iodine treated (in either order) with 10 c.m.³ of a saturated solution of potassium bicarbonate and 10 c.m.³ of decinormal iodic acid gave the same distinctive colour to chloroform as came from the same amount of iodine in the absence of the iodic acid. So it appears that if in the system under consideration, reactions do occur between iodic acid and iodine to alter the amount of iodine recoverable, such action is not appreciable between small amounts of these materials. This, however, does not preclude the possibility of perceptible changes under the mass-action of a large amount of iodine.

The reactions of hydrochloric acid, and probably of hydrobromic acid, in the presence of varying amounts of iodic acid, iodine, and iodide, as well as the reaction of the alkaline carbonate upon such mixtures, are doubtless complex, more or less reversible, and dependent upon proportion and dilution. The tendency of the former reactions is toward the reduction of the molecule of iodic acid, and the formation of the chloride or bromide of iodine. Thus, Miss Roberts (*loc. cit.*) demonstrated that a solution of hydrochloric acid, so dilute that by itself it is without effect on iodic acid, acts upon a mixture of iodic acid with either free iodine or an iodide to form iodine monochloride. The action of the acid carbonate upon the iodine chloride or bromide may produce a salt of the oxy-acids and free iodine.

The practical effects, under the conditions of analysis, of the reaction between iodine, iodic acid, and the halogen acids in presence of sulphuric acid, and of reactions which may occur upon neutralisation by an acid carbonate, were studied in detail in a number of experiments.

The preliminary experiments of Table I. were made to bring out the effect of neutralising with the acid carbonate and subsequently titrating with an alkaline arsenite a solution containing sulphuric acid and a considerable amount of free iodine. The danger of mechanical loss of iodine during the effervescence accompanying neutralisation, as well as by spontaneous volatilisation from the surface during the process of titration, was minimised by effecting the neutralisation in the trapped Drexel washing

* The method of preparation of a sample for analysis in the case of the more brittle forms of iron, by crushing in a steel mortar and sifting, is suggested in Regnault's "Elements of Chemistry," translated from the French by Beiloin, 1867, ii., 112.

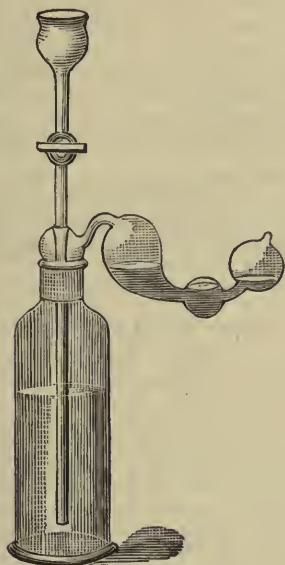
† Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, Fourth Series, vol. iii., No. 16.

TABLE I.
Effect of the Carbonate.

[5 c.m.³ H₂SO₄ (1:3). Total volume of liquid, 250 c.m.³.]

I (in KI) taken. Grm.	KHCO ₃ in excess. C.m. ³ .	I found. Grm.	Error. Grm.
1. 0.0713	Very small	0.0707	0.0006—
2. 0.0715	Very small	0.0710	0.0005—
3. 0.0713	10	0.0710	0.0003—
4. 0.0710	10	0.0706	0.0004—
5. 0.0723	10	0.0717	0.0006—
6. 0.0713	20	0.0709	0.0004—
7. 0.0713	20	0.0709	0.0004—
8. 0.3565	Very small	0.3560	0.0005—
9. 0.3568	Very small	0.3561	0.0007—
10. 0.3567	10	0.3563	0.0004—
11. 0.3596	10	0.3588	0.0008—
12. 0.3565	10	0.3565	0.0000
13. 0.3572	20	0.3560	0.0012—
14. 0.3567	20	0.3569	0.0002+

bottle, to be described later, and making the titration in the same tall washing cylinder without transfer. To varying amounts of a recently standardised decinormal solution of iodine were added successively 5 c.m.³ of dilute sulphuric acid and varying amounts of potassium bicarbonate in excess of that necessary to neutralise the free acid, decinormal arsenious acid in slight excess of the



iodine, 5 c.m.³ of starch emulsion, and decinormal iodine to colouration, the total volume of the liquid being not greater than 250 c.m.³. The results show plainly that while the loss, mechanical or otherwise, in the treatment of reasonably large amounts of fairly concentrated iodine is perceptible, it is still well within permissible limits (amounting to a little less than 0.0005 grm. in the mean), and obviously independent of the excess of the carbonate in the solution, and of the amount of free iodine present.

In the experiments of Table II. the proposed process of analysis was tested upon potassium iodide taken by itself in varying amounts of a $\frac{1}{10}$ normal solution and carefully standardised by the method formerly elaborated in this laboratory (Gooch and Browning, *Am. Journ. Sci.*, xxxix., 188). The apparatus employed was a Drexel washing bottle of 500 c.m.³ or 1000 c.m.³ capacity, according to requirements, with stopcock and thistle-tube fused to the inlet-tube and a Will and Varrentrapp absorption-trap sealed to the outlet, as shown in the accompanying

figure. The iodide for the test was drawn from a burette into the bottle and carefully washed down, and potassium iodate in excess of the amount theoretically necessary (namely, 5 c.m.³ of a 0.5 per cent solution for every portion of 20 c.m.³ of the iodide solution), was added, and the volume of the liquid was adjusted to the volume at which it was desired that the iodic and hydriodic acids should react. The stopper with the thistle-tube and trap was now placed on the bottle and the trap was half-filled by means of a pipette with a 5 per cent solution of potassium iodide. Five c.m. of dilute (1:3) sulphuric acid were added through the thistle-tube and washed down; the stopcock was closed, and the solution gently agitated, if necessary, to insure a complete separation of iodine. Potassium bicarbonate in saturated solution to an amount about 10 c.m. in excess of that required to neutralise 5 c.m.³ of dilute (1:3) sulphuric acid, was poured into the thistle-tube, and allowed to flow into the bottle slowly enough to avoid a too violent evolution of gas. The stopcock was closed, and the solution agitated by giving to the bottle a rotary motion, at the same time keeping the bottom pressed down upon the work table, to prevent a possible splashing of the iodide out of the trap into the yet acid solution. When the neutralisation of the solution had been completed, the bottle was shaken until the last trace of violet vapour was absorbed in the liquid. The greater part of the solution in the trap was then run back into the bottle, the stopper removed, and the tube and trap carefully washed; the washings being added to the bulk of the solution. Decinormal arsenious acid was introduced from a burette to the bleaching point, 5 c.m.³ of starch emulsion were added, and the solution was titrated back with decinormal iodine (usually only a few drops) to colouration.

TABLE II.
Effect of Dilution.

KI taken. Grm.	KI found. Grm.	Error. Grm.	Approximate volume upon addition of H ₂ SO ₄ . C.m. ³ .	Volume H ₂ SO ₄ (1:3) used. C.m. ³ .
1. 0.0772	0.0768	0.0004—	150	5
2. 0.0772	0.0765	0.0007—	150	5
3. 0.1544	0.1546	0.0002+	150	5
4. 0.1544	0.1541	0.0003—	150	5
5. 0.3087	0.3090	0.0003+	150	5
6. 0.3087	0.3088	0.0001+	150	5
7. 0.3859	0.3864	0.0005+	150	5
8. 0.3859	0.3860	0.0001+	150	5
9. 0.0772	0.0754	0.0018—	300	5
10. 0.0772	0.0757	0.0015—	300	5
11. 0.1543	0.1532	0.0011—	300	5
12. 0.1544	0.1524	0.0020—	300	5
13. 0.0772	0.0744	0.0028—	500	5
14. 0.0772	0.0737	0.0035—	500	5
15. 0.1544	0.1521	0.0023—	500	5
16. 0.1544	0.1512	0.0032—	500	5
17. 0.3859	0.3827	0.0032—	500	5
18. 0.3859	0.3831	0.0028—	500	5
19. 0.0772	0.0744	0.0028—	500	10
20. 0.0772	0.0757	0.0015—	500	10
21. 0.3859	0.3828	0.0031—	500	10
22. 0.3859	0.3827	0.0032—	500	10

Blank tests made upon a solution obtained by mixing the maximum amount of the iodate with 5 c.m.³ of dilute sulphuric acid (1:3), neutralising as usual with potassium bicarbonate, adding the iodide from the trap and 5 c.m.³ of starch emulsion, showed that a single drop of iodine was invariably sufficient to bring out the starch blue. Occasionally it was found that the mixture, particularly when chlorides or bromides were present, of itself developed a trace of colour, but by no means a reading tint. A correction of the one drop of iodine necessary to

bring out the colour reaction in the blanks was applied uniformly in the analytical process.

The number of centimetres of decinormal arsenious acid required to bleach the free iodine, multiplied by 0.01383 (log. 2.140822) gives the number of grms. of potassium iodide taken for analysis, being equivalent to five-sixths of the iodine liberated in the solution.

From these results it appears that the degree of dilution of the solution at the time when the mixed iodide and iodate are acidified has an important influence on the completeness of the reaction. Thus, the mean error of the determinations in which the volume at the time of the reaction did not exceed 150 c.m.³ was practically nothing, while the errors at volumes of 300 c.m.³ and 500 c.m.³ amounted to 0.0016 grm. and 0.0028 grm. respectively. It is obvious that the doubling of the amount of sulphuric acid used in acidifying does not increase the amount of iodine liberated at the highest dilution. The plain inference is that the interaction between the iodide and iodate should be brought about in a volume of liquid not much exceeding 150 c.m.³.

In the following series of experiments, recorded in Table III., the effect of the introduction of a chloride or bromide into the iodide (before the iodate is added) was studied. The volume of the liquid at the time of acidifying was fixed at 150 c.m.³, approximately, and 5 c.m.³ of the dilute sulphuric acid (1:3) were used. The mode of procedure was otherwise similar to that of the foregoing series.

TABLE III.

Effect of Chloride and Bromide.

	KI taken. Grm.	KI found. Grm.	Error. Grm.	NaCl taken. Grm.	KBr taken. Grm.
1.	0.0772	0.0795	0.0023+	0.2	—
2.	0.0772	0.0784	0.0012+	0.2	—
3.	0.0771	0.0823	0.0052+	0.5	—
4.	0.0773	0.0819	0.0046+	0.5	—
5.	0.1544	0.1588	0.0044+	0.5	—
6.	0.1544	0.1590	0.0046+	0.5	—
7.	0.0772	0.0802	0.0030+	—	0.2
8.	0.0773	0.0853	0.0080+	—	0.2
9.	0.0772	0.0873	0.0101+	—	0.5
10.	0.0772	0.0861	0.0089+	—	0.5
11.	0.1544	0.1646	0.0102+	—	0.5
12.	0.1543	0.1626	0.0083+	—	0.5

The influence of sodium chloride and potassium bromide in increasing the amount of iodine liberated is plain. The increase comes without doubt from the iodate, and is doubtless due to the formation of iodine chloride or bromide, during the acidifying, by the interaction of the free iodine, the iodic acid, and the hydrochloric or hydrobromic acid, according to the reactions previously discussed. It is plain, therefore, that the value of the process in the determination of iodine in an iodide is restricted of necessity to those cases in which it is known that chlorides or bromides are not present to any considerable extent. For determining the standard of a solution of nearly pure potassium iodide, employed in so many laboratory processes, it should find useful application.

In Table IV. are comprised a number of experiments made exactly like those which seemed to give the best results in the series of Table II. The iodide and an excess of iodate (5 c.m.³ of the 0.5 per cent solution to every 20 c.m.³ of N/40 iodine) were made to interact in a volume of about 150 c.m.³, 5 c.m.³ of sulphuric acid (1:3) were used to bring about the reaction, 10 c.m.³ of potassium bicarbonate were added after the neutralisation of the sulphuric acid was complete, and the free iodine was estimated by titrating decinormal arsenious acid, the manipulation being like that previously described in detail.

The average result of a series of several determinations in which a great excess (0.1 grm.) of potassium iodate was used, proved to be practically identical with that of a

TABLE IV.

Analysis of Pure Potassium Iodide.

	KI taken. Grm.	KI found. Grm.	Error. Grm.
1.	0.0814	0.0816	0.0002+
2.	0.0814	0.0813	0.0001—
3.	0.0814	0.0805	0.0009—
4.	0.0815	0.0809	0.0006—
5.	0.0814	0.0808	0.0006—
6.	0.0814	0.0806	0.0008—
7.	0.0814	0.0812	0.0002—
8.	0.1628	0.1624	0.0004—
9.	0.1628	0.1617	0.0011—
10.	0.1628	0.1621	0.0007—
11.	0.1628	0.1619	0.0009—
12.	0.1628	0.1624	0.0004—
13.	0.1628	0.1621	0.0007—
14.	0.1628	0.1626	0.0002—
15.	0.2442	0.2451	0.0009+
16.	0.2442	0.2442	0.0000
17.	0.2442	0.2439	0.0003—
18.	0.3256	0.3258	0.0002+
19.	0.3256	0.3256	0.0000
20.	0.3256	0.3258	0.0002+
21.	0.3256	0.3272	0.0016+
22.	0.3256	0.3256	0.0000
23.	0.4071	0.4076	0.0005+
24.	0.4071	0.4080	0.0009+
25.	0.4071	0.4073	0.0002+

similar series in which only a small excess of the iodate was employed, so that it appears to be unnecessary in any practical work to restrict the amount of iodate below the amount necessary to decompose the maximum quantity of potassium iodide which we have handled, namely, 0.4 grm.

It appears that for the estimation of iodine in a soluble iodide free from notable amounts of chlorides or bromides, this method, depending as it does upon a single standard solution, is simple, fairly accurate, and rapid.

SODIUM PEROXIDE AS A THIRD GROUP REAGENT.

By S. W. PARR.

Sodium peroxide as a reagent has properties of a very unusual and striking character. These properties are no less valuable than peculiar, and indicate for this substance a prominent place in analytical work. The immediate object of this paper is to note the advantages and adaptability of sodium peroxide to qualitative analysis. By this means its numerous characteristics can be best illustrated. The specific data indicating its use in certain lines of quantitative analytical work will be given later. The methods herein set forth have been employed in this laboratory during the past year by large classes in qualitative analysis. This practical test of the processes involved has abundantly demonstrated their value.

In the ordinary procedure for the separation of the metals the greatest difficulty arises in the third or iron group. These complications may be briefly enumerated as follows:—

(a). The separation of zinc in the presence of chromium.

(b). The unsatisfactory separation of cobalt and nickel from the other members of the group by the action of dilute hydrochloric acid on their sulphides.

(c). The variations arising from the presence of phosphates, &c.

It is not necessary to enlarge upon these difficulties. The one most commonly ignored in methods is usually

outlined, and yet a very serious obstacle is the one designated under (a). Zinc and chromium enter into a combination which, to a very large extent, resists the action of ammonia and ammonium salts. The use of barium carbonate to obviate this difficulty is cumbersome. By use of sodium peroxide we may oxidise the chromic compounds present to sodium chromate, and thus completely eliminate it as a factor in any precipitation likely to be employed, excepting of course such as would involve a reduction and return to the condition of a chromic salt.

The method of procedure is as follows:—The solution should be slightly acid. A small porcelain spoonful of the peroxide is slowly sifted in with constant stirring. The solution is then heated to complete the decomposition of the peroxide, and finally boiled for some minutes after the oxygen seems to be all driven off. The completeness of the oxidation may be easily tested by filtering from any insoluble constituents, acidifying, boiling, and making ammoniacal. A precipitate may be aluminum or unoxidised chromium. Filter and wash free from all sodium chromate, re-dissolve in a little nitric acid, and treat as before with a small amount of sodium peroxide. A yellow colouration is due to the chromium which escaped oxidation by the first treatment. However, if properly conducted, the first operation should be complete. Similarly, the insoluble residue on the filter, if suspected of being a zinc-chromium compound, may be washed free from chromate, dissolved in dilute nitric acid, and treated again with the peroxide. The only condition so far governing the completeness of the transformation to the chromate form is the necessity of starting the oxidation with the chromium entirely in solution. Precipitated chromium hydroxide will undergo this transformation, but less readily, and especially if the precipitate is the double one of zinc and chromium. Hence the advisability of beginning the oxidation with the solution containing some free acid, preferably nitric. The quantity of free acid is immaterial, less than 1 c.c. being sufficient. It should be noted, however, that the amount of sodium peroxide should cause the solution to pass quite beyond the neutral condition, since the oxidation is only partial while in the acid state. It might be expected that the moment the addition of sodium peroxide passed the neutral point the precipitation of chromium would commence, and the completeness of the oxidation be lessened in consequence, but I have not found this to be the case. The oxidising action of the peroxide is so pronounced that it precedes the precipitating action, hence the reason for using the dry sodium peroxide. A cold saturated solution of the peroxide will operate but incompletely. Hydrogen peroxide will also act similarly, but even less completely than the solution of sodium peroxide. For obvious reasons also the operation is performed on the solution before heating, and it is better to shake the powder in gradually than to drop the reagent in at once.

We are ready now to note the effect of such treatment as above indicated, upon the other members of this group, assuming that any or all may be present, including cobalt and nickel. The results are as follows:—

(a). Aluminum compounds are in solution in the form of sodium aluminate, not different from the ordinary result from using sodium hydroxide in excess. It is assumed, of course, that the sodium peroxide has exceeded the free acid in sufficient amount to provide sodium hydroxide in excess.

(b). Zinc is similarly in solution as zincate.

(c). Iron precipitates as a very dense, reddish brown precipitate, the exact composition of which is being made a matter of investigation. The precipitation is complete, no re-solution being effected upon boiling. The filtration is performed with great facility. The precipitate is almost insoluble in concentrated nitric acid; soluble in dilute acids on heating. If phosphates are present none are precipitated with the iron, but all pass through and are found in the filtrate.

(d). Manganese behaves exactly as iron, precipitating presumably as the hydrated dioxide, $\text{MnO}_2 \cdot \text{H}_2\text{O}$, having all the properties of that compound as to colour, solubilities, &c. Similarly also phosphates are not precipitated. It should also be noted that from this precipitate can most readily be obtained the delicate test for the presence of manganese by formation of permanganic acid by means of nitric acid and lead peroxide or Pb_2O_4 .

(e). Cobalt precipitates also a black hydrated cobaltic oxide with solubilities the same as in the case of iron and manganese. No phosphate is precipitated with the cobalt. The precipitate, in conjunction with dilute acid and potassium iodide, liberates free iodine, imparting an intense blue to starch solution. This latter property, however, is common to the precipitates of iron and manganese under (c) and (d).

(f). Nickel precipitates, as the ordinary green nickelous hydroxide, $\text{Ni}(\text{OH})_2$, easily soluble in acid, either concentrated or dilute. As to phosphates, in the case of nickel, if present in large amounts, small quantities are found in the precipitated nickel. A re-solution and re-precipitation with sodium peroxide, however, eliminates all the phosphate from the precipitate. The behaviour of nickel in thus precipitating as the nickelous compound indicates for its higher form of oxidation a less degree of stability than exists in the case of cobalt. This property suggests the readiest and most delicate method for the detection of nickel, even in the presence of the three precipitates enumerated above, thus:—Boiling this precipitate of nickel with bromine water converts it at once into the black nickelic hydroxide, which has the property of decomposing potassium iodide with water alone, no acid being required, as in the case of iron, manganese, and cobalt. It is necessary, of course, to boil off the free bromine, which is readily accomplished. The action upon a potassium iodide starch solution is very marked.

The above facts suggest a method for the iron group which is indicated by the accompanying table. It is given here to illustrate the adaptability of some of the well-known but more positive and satisfactory tests for the several metals.

In the presence of phosphates the method so far employed has been as follows:—Upon dissolving the precipitate from the ammonium sulphide in concentrated nitric acid, a very little of the solution is tested for phosphoric acid in the usual manner. If present, granulated tin is added and the boiling continued. Filter from the insoluble tin phosphate, make ammoniacal, re-precipitate with ammonium sulphide, and proceed as with phosphoric acid absent. Any method not involving the use of tin, and depending upon the non-formation of the phosphates of iron, manganese, and nickel, is as yet unsatisfactory. Having removed the barium and strontium with sulphuric acid before the precipitation with ammonium sulphide, the oxidation and precipitation by means of sodium peroxide may be performed as usual; but before filtering, the solution is made acid with acetic acid, and boiled a little further and filtered. The filtrate now may contain, besides the aluminum, zinc, and chromium, the nickel which is readily soluble in acetic acid, and the calcium and magnesium which has been brought along by means of the phosphoric acid. A little of the cobalt, however, dissolves with the acetic acid, and a solvent has not been found thus far for the calcium phosphate and nickel hydroxide that will not dissolve traces of the other three metals of the precipitate.

One other application to qualitative analysis may be mentioned as having proved valuable. In testing for acids a ready method for distinguishing between carbon dioxide and sulphur dioxide, when both are present, is found in the use of a solution of sodium peroxide. Conducted into this solution, the above gases form sodium carbonate and sodium sulphate respectively. With lime-water the solution will give a copious precipitate if the carbonate has been formed, and with an acidulated solution of barium chloride the sulphate test is obtained.

The precipitate obtained in the ordinary method by means of ammonium hydroxide and ammonium sulphide contains, as hydroxides and sulphides, iron, manganese, cobalt, nickel, aluminum, zinc, and chromium, and is brought into solution by means of 10 to 15 c.c. of concentrated nitric acid with heat. Nearly neutralise with sodium hydroxide, then silt in slowly with stirring sodium peroxide in excess. Boil.

Precipitate A contains iron, manganese, cobalt, and nickel.

(a). Test for iron by dissolving a small portion of the precipitate in dilute hydrochloric acid and adding potassium thiocyanate. The blood-red colouration is due to ferric thiocyanate.

(b). For manganese, to 5 c.c. of water add 5 c.c. concentrated nitric acid and 5 to 10 grms. Pb_3O_4 . Stir into the warm mixture a little of the precipitate and let stand. A purple solution is permanganic acid.

(c). In absence of iron or manganese, stir a little of the precipitate into dilute hydrochloric acid, and add solution of potassium iodide and starch. In presence of iron and manganese use the bead test.

(d). Boil some of the precipitate with bromine water till all bromine is expelled, add water and solution of potassium iodide and starch, $Ni(OH)_3 + KI = Ni(OH)_2 + KOH + I$, imparting the blue to the solution.

Solution A contains all the aluminum, zinc, and chromium. The yellow colour is evidence of chromium. Acidify with hydrochloric acid, boil, and add ammonium hydroxide.

Precipitate B consists of aluminum hydroxide, and any chromium hydroxide that may have escaped oxidation. Dissolve in nitric acid, and repeat the precipitation with sodium peroxide, or apply the blowpipe and cobalt test for aluminum.

Solution B contains zinc and chromium.

(a). Test for zinc by adding to a portion a few drops of potassium ferrocyanide. A heavy white precipitate indicates zinc.

(b). If further verification of chromium is needed, make the solution acid with hydrochloric acid, and boil with a little alcohol added. The chromium reverts to the green chromic chloride.

Many other features incidental to the properties above outlined have developed, mainly of interest in quantitative methods. It is hoped that the data will be of sufficient value to warrant further notice.—*Journal of the American Chemical Society*, xix., p. 347.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING MARCH 31ST, 1897.

By WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolis Water Act, 1871.

London, April 10th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 189 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from March 1st to March 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in a previous report.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 189 samples examined one was recorded as "turbid," the remainder being clear, bright, and well filtered.

There has been a large excess of rain recorded at Oxford during the month, the actual fall being 2.61 inches; as the average fall for thirty years is only 1.50 inches, we have had an excess of 1.11 inches.

Our bacteriological examination of the London waters gives the following results:—

	Microbes per c.c.
Thames water, unfiltered (average of 27 samples)	9187
Thames water, from the clear water wells of five Thames-derived supplies (average of 159 samples)	33
Ditto ditto highest	372
Ditto ditto lowest	2
New River, unfiltered (average of 27 samples)	1160
New River, from the Company's clear water well (average of 27 samples)	30
River Lea, unfiltered (average of 27 samples)	1080
River Lea, from the East London Water Company's clear water well (average of 27 samples)	22

Last month we drew attention to the abnormal bacterial contents of some samples taken from the wells of the Grand Junction Water Works at Hampton, connected with the supply of the country district. The Engineer has now informed us that the Company in May last authorised extensive additions and alterations to their filtering plant at Hampton; these are in course of construction. The Board, however, having had our recent communications on the subject brought to their notice, have now authorised the Engineer to carry out more extensive alterations than had been previously contemplated. We have had a consultation with the Engineer, at which he submitted the general plans of the proposed new filtering plant, which in our opinion will meet the difficulty.

Since the beginning of the month the water from the Hampton Works has steadily improved, and for the last ten days it has been in a satisfactory condition.

At this season of the year there is always a considerable amount of fish spawn in the river, clogging up the filters, and rendering very frequent cleaning necessary. This, added to the recent heavy rainfall, has put a severe strain on the filtration plant of the different Companies. The results, however, show that they have been well able to cope with the difficulties.

We are, Sir,

Your obedient Servants,
WILLIAM CROOKES,
JAMES DEWAR.

THE HYDROLYSIS OF ACID AMIDES.

By IRA REMSEN.

A NUMBER of years ago, with the assistance of advanced workers in this laboratory, I carried out a series of investigations on the oxidation of substitution-products of aromatic hydrocarbons, the results of which went to show that, when the oxidising agent is chromic acid, an oxidisable residue, situated in the ortho position with reference to an atom or group that is not oxidisable, is almost completely protected from oxidation, whilst similar groups in the meta or para position are easily oxidised. Later, experiments were tried on the reduction of nitro compounds of different structure, but, although results of some value

were thus obtained, these have not been published, as I have hoped that the method of making the measurements might be improved. Still later, my attention was drawn to the marked difference between benzoic sulphinide (saccharin) and parasulphaminebenzoic acid (Remsen and Burton, *Am. Chem. Journ.*, xi., p. 403) towards boiling dilute acids. The former is easily converted into the corresponding acid ammonium salt, while the latter remains unchanged.

About a year ago I requested Mr. E. E. Read, of this laboratory, to make some experiments on the relative ease with which the three nitrobenzamides are converted into their ammonium salts by dilute acids. Mr. Read has since devoted himself with much skill and energy to this work, and the results reached are most interesting. A method has been devised by which it is possible to measure with a considerable degree of accuracy the rate of the hydrolysis. The experiments thus far completed have been carried out with hydrochloric acid of three concentrations and with sulphuric acid of three concentrations. The details of the experiments will be communicated later, and it will then be seen that the curves showing the rate of change are remarkably regular. A striking difference is shown in the action of the three nitrobenzamides. The following results obtained with half normal hydrochloric acid may serve as an example:—

Time in hours.	Per cent of o-amide changed.	Per cent of m-amide changed.	Per cent of p-amide changed.
$\frac{1}{2}$	—	21.1	24.4
1	—	42.8	48.2
$1\frac{1}{2}$	—	50.1	63.4
2	—	65.0	71.2
3	3.3	80.5	84.5
4	3.9	88.9	91.9
5	—	92.6	94.6
6	6.2	94.2	96.9
7	—	—	—
8	8.6	—	—

The orthoamide is seen to resist the action of the hydrolysing agent to a very marked degree, so that it was difficult to measure the amount of the change if the heating was not continued for at least three hours. On the other hand, the meta and para amides yield readily, the para somewhat more so than the meta.

It seems highly probable that other aromatic acid amides that contain an atom or group in the ortho position to the group CONH_2 will conduct themselves in a similar way. These phenomena are suggestive of those studied in this laboratory many years ago, to which reference has already been made. Apparently the single nitro group in the ortho position with reference to the carbamide group, CONH_2 , protects this from the action of dilute acids, as the nitro group in the ortho position to methyl protects this from oxidation by chromic acid, as shown in my earlier experiments. There is also some analogy between these protection phenomena and those recently studied by Victor Meyer and his students—an analogy which Meyer does not appear to have recognised.

The object of this note is to inform chemists that we are in possession of a method that makes measurements of the kind given above comparatively easy, and that we propose to apply the method to the study of as large a number of cases as possible, with the object of determining—

1. Whether the influence of ortho groups upon the hydrolysis of acid amides is always the same; and
2. How various atoms and groups differ in their effect upon the rate of hydrolysis.

This work will require some time, and it seems best to postpone the publication of the results until the investigation is completed.

(The substance of this note was communicated by me to the National Academy of Sciences, at the New York

meeting held November 18, 1896).—*American Chemical Journal*, April, 1897.

WHO SHALL BE HEN-WIFE.

"Na ye maun gan wi' me the noo, Wullie" (his name was Charles), said Henrietta, as she flung a brawny arm round the lad's neck; "sure my friend Jemmie's as Scotch as they make 'em."

"Na, na, lass; I maun gan wi' Wilhelmina, an' my name's no Wullie,—hang it l!—and ye need na gar me a crick in my—what do you call it in this lingo?—craig. Your fren' Jemmie's a fause loon; didna she deceive the kirk and get the mickle rebuke o' the meeneester?"

"Then ye'll no mair keep company wi' me (hang this jargon!)" said Henrietta, who was English, and found Kailyardish trying.

Said Charles, "I care no for ye, an I ne'er lo'ed ye nor e'r any but the Scots lassie Wilhelmina."

"You're a rude little boy," said she.

"Stay y're havers, and no clack like siccan," said the Chem. Soc., afflicted by the *genius temporum*, and involuntarily speaking the lingo.

"An' why shall we no have a bit clavers? Sair, I ken I'm as winsome as Jemmie," said Wilhelmina, as she tried to pull Wullie to her side.

"Bide a wee," said a voice. It was Jemmie's.

There was a confused struggle; Wullie squealed and kicked, the Chem. Soc. awoke with a start, and found Jemmie installed as hen-wife, and the kailyard resuming its normal gentlemanly demeanour.

IAN McCROCKETT.

EDINBURGH UNIVERSITY GRADUATION CEREMONIAL.

PROFESSOR SIR LUDOVIC GRANT, Bart., Dean of the Faculty of Law, introduced Professor James Dewar, F.R.S., Royal Institution, London, to receive the degree of LL.D.

The University recalls with pride that the distinguished physicist and experimentalist who now stands before us received his earliest training in science and first impulse towards research within her precincts. For Professor Dewar was a pupil of Professor Tait's, and thereafter acted as assistant to Professor, now Lord, Lyon Playfair, when he held the Chair of Chemistry in Edinburgh. Since that period Professor Dewar has been called to many offices which are the prerogative of the highest scientific eminence. He was a member of the Government Committee on Explosives, and of the Royal Commission on the Metropolitan Water Supply, and is now President of the Chemical Society, Jacksonian Professor of Natural Experimental Philosophy in the University of Cambridge, and Fullerian Professor of Chemistry in the Royal Institution. But a bare recital of his appointments conveys no adequate idea of Professor Dewar's services to science. Despite the duties of official life, he has devoted himself untiringly to experimental research, and his investigations have been productive of the most remarkable results, and constitute his chief claim to academic recognition. In particular may be mentioned the discoveries he has made regarding the liquefaction of gases, and the properties of matter at a very low temperature. In many instances his experiments demanded the highest courage, no less than perseverance and skill, for they were fraught with extreme personal danger. His Alma Mater rejoices to follow the example already set by the Universities of St. Andrews and Glasgow, by conferring upon her distinguished alumnus the honorary degree of Doctor of Laws. (Applause).—*Scotsman*, April 12, 1897.

NOTICES OF BOOKS.

An Outline of the Theory of Solution and its Results. For Chemists and Electricians. By J. LIVINGSTON R. MORGAN, Ph.D. (Leipzig), Instructor in Quantitative Analysis, Polytechnic Institute, Brooklyn (New York). New York: John Wiley and Sons. London: Chapman and Hall, Limited. 1897. 63 pp., 12mo.

THIS little book should be in the hands of every student of chemistry who cares to make himself acquainted with recent developments in chemical philosophy, and every student who does *not* care should be required to pass an intelligent examination on its sixty-three pages.

The author has compiled a very clear, condensed, and up-to-date account of the modern theory of solution and its influence on chemical philosophy; he shows its development logically and historically as based on electrolytic dissociation, from the laws governing the behaviour of gases. The results obtained by van't Hoff, Arrhenius, and Ostwald (to whom the book is dedicated) are admirably stated, and without being overloaded with mathematical expressions. Students of analytical chemistry who have not the time to digest the larger treatises of Ostwald and Le Blanc, will find this little work valuable in removing the empiric character of their studies and processes; the laboratory worker will learn *why* an excess of a precipitant is efficacious, and *why* certain salts are added to water used in making precipitates, and *why* indicators in volumetric analysis act as they do.

The theory of solution is daily becoming more and more important for theoretical and practical chemistry, as well as for electricity, and Dr. Morgan has supplied an excellent introduction to the larger treatises.

One typographical error cannot be overlooked: Helmholtz appears twice as "Helmholst," but the book is neatly printed.

H. C. B.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 14, April 5, 1897.

Preparation of Iron Carbide by the Direct Union of the Metal and of Carbon.—Henri Moissan.—If we heat pure iron and the carbon of sugar to the high temperature of the electric furnace, and then allow the regulus to cool slowly, we find in the metal merely a small trace of combined carbon. We thus obtain a grey casting which solidifies towards 1150°. If the metal is run into a mould at the temperature of 1300° to 1400° it contains on cooling graphite and a larger quantity of combined carbon; this is white cast metal. Lastly, if we cool abruptly in water iron saturated with carbon at 3000°, there is produced an abundant crystallisation in the metal, and we may separate from it a pure definite crystalline carbide, CFe_3 . This carbide is identical with that of steel. All these facts are explained simply on admitting that iron carbide, like ozone and silver oxide, may be formed at a very high temperature and then be decomposed progressively on a reduction of temperature. We find a notable quantity in steel, the melting-point of which is high, rather less in white cast metal, and very little in grey castings. In all our experiments we have observed the formation of the carbide only in the liquid metal.

Nomination.—The Academy proceeded to the nomination of a member in the Section of Astronomy, *vice* M. Tisserand, deceased. M. Radau obtained the absolute majority of the votes, and was accordingly proclaimed elected.

A letter was read from H. Wilde, F.R.S., President of the Manchester Literary and Philosophical Society, addressed to M. Berthelot, criticising as improper the expression the "Periodic Law," and offering to the Academy the sum of £5500 (=137,500 frcs.) to be invested in French securities and the interest applied to the foundation of an annual prize of 4000 frcs. to be awarded to the author of any discovery or research in astronomy, physics, chemistry, mineralogy, geology, and mechanics, which in the judgment of the Academy shall be considered the most meritorious. The award of this prize will be international, and may be retrospective. (See CHEMICAL NEWS, vol. lxxv., p. 177).

Partial Polarisation of the Radiations emitted by some Sources of Light under the Influence of the Magnetic Field.—N. Egoroff and N. Georgiinsky.—Some months ago Dr. Leeman, of the University of Leyden, made remarkable experiments on the influence of a fairly strong magnetic field on the emission of flames in a Bunsen Burner (sodium and lithium). He demonstrated that the perturbation undergone by the ions under the influence of the magnetic focus produce new periods and luminous vibrations (expansion of the spectral rays of sodium and lithium). Subsequently Dr. Leeman, led by the theoretic views of Prof. Lorentz, has demonstrated the peculiar polarisation of these new vibrations.

New Cadmium Lamp for the Production of Interference Fringes.—Maurice Hamy.—The resistance of the lamp when in action is equal to that of one-fifth m.m. of air. A decided rise of temperature above 350° increases this resistance, so that the discharge no longer takes place in the apparatus. Besides the four radiations—red, green, blue, indigo—the spectroscopist has enabled me to identify all the known visible rays of cadmium in the light given out by the lamp; and besides a faint ray in the red (λ 632), the rays of sodium, a fine ray in the green (λ 515), invisible in the spectrum of the spark, striking into the air between the electrodes of cadmium.

Researches on Nickel Steels. Meteorological Properties.—Ch. E. Guillaume.—A mechanical paper.

Nature of various kinds of Radiations produced by Bodies under the Influence of Light.—G. Le Bon.—This paper will be inserted as early as possible.

New Oxide of Phosphorus.—A. Besson.—The composition of this phosphorous oxide is shown by the formula P_2O .

Metastannyl Chloride.—R. Engel.—This paper will be inserted in full.

Action of High Temperatures upon Copper, Bismuth, Silver, Tin, Nickel, and Cobalt Sulphides.—A. Mourlot.—We can completely desulphurise the bismuth and copper sulphides, but the desulphuration of bismuth is by far the more difficult. Silver sulphide on exposure to the highest temperatures yields a volatile product still retaining traces of sulphur. Cobalt and nickel yield sulphides relatively stable, CoS and Ni_2S . Tin sulphide undergoes a partial volatilisation and furnishes a crystalline regulus of protosulphide, a new example of a sulphide stable at a high temperature.

Combinations of Gaseous Ammonia and Methylamine with the Haloid Compounds of Lithium.—J. Bonnefoir.—A thermo-chemical paper.

Action of Tannin and of Gallic Acid upon certain Alkaloids.—Oeschner de Coninck.—The author examines the action of dry pulverulent tannin upon pure nicotine and nicotine in aqueous solution. The result was negative, as also in the case of alcoholic and ethereal solution.

Preparation of Sodium Carbide and Mono-sodic Ethylene.—Camille Matignon.—The author describes the *modus operandi* which he has adopted.

Observations Concerning the Freezing Temperature of Milk.—J. Winter.—A controversial paper in reply to MM. Bordas and Génin.

Non-identity of the Lipases of Different Origins.—M. Hanriot.—The blood of the eel contains the same lipase as the blood of the horse, but in a much greater quantity.

Certain Properties of the Ferment of Fracture of Wines.—P. Cazeneuve.—The author enquires into the action of sulphurous acid, which in a small proportion destroys the action of an oxidase, and thus prevents the "fracture" of wines.

Novel Method of obtaining the Perfume of Flowers.—Jacques Passy.—The author uses water in place of the fats used in the process of *enfleurage*.

Bulletin de la Société d'Encouragement pour l'Industrie Nationale. Series 5, Vol. ii., No. 2.

Economical Treatment of the By-products of Distillation of Starchy Products.—A report presented on behalf of the Committee of Chemical Arts by M. de Luynes on the procedures adopted by MM. Bonard and Boulet, who, when treating the distillery residues by the procedure described and figured in this paper, utilise them for the production of oils and matters suitable for cattle-foods.

Revue Universelle des Mines et de la Metallurgie. Series 3, Vol. xxxvii., No. 3.

This issue contains no chemical matter.

MISCELLANEOUS.

Tuberculin O and R.—Prof. Koch has succeeded in the production of two new preparations which he names as above, both of which possess an immunising action against the bacilli of tuberculosis. The preparation is injected beneath the skin at first in small quantities, and the dose is then gradually increased.

Test for Formaldehyd.—L. Kentmann (*Pharm. Gen. Anz.*, 1896, viii., 356).—If the suspected liquid is floated on an equal volume of a solution of 0.1 grm. of morphine hydrochloride in 1 c.c. of strong sulphuric acid, a red violet colour is produced within a few minutes, provided the formalin exceeds one part per 6000.—*The Analyst*.

MEETINGS FOR THE WEEK.

TUESDAY, 27th.—Royal Institution, 3. "Volcanoes," by Dr. Tempest Anderson, B.Sc.
Society of Arts, 8. "Delft Ware," by Dr. J. W. L. Glaisher, F.R.S.

WEDNESDAY, 28th.—Society of Arts, 8. "Asbestos and Asbestos—with some Account of the Recent Discovery of the latter at Danville, in Lower Canada," by Robert H. Jones.

THURSDAY, 29th.—Royal Institution, 3. "Liquid Air as an Agent of Research," by Prof. Dewar, F.R.S., &c. (The Rev. Canon Ainger being unable through illness to begin his lectures on this day).

Chemical, 8. "Monochloridiparacetic Acid and some Condensations," by H. C. Myers, Ph.D.
"Decomposition of Iron Pyrites," by W. A. Caldecott, B.A.

FRIDAY, 30th.—Royal Institution, 9. "Cathode Rays," by Prof. J. J. Thomson, F.R.S., &c.

SATURDAY, May 1st.—Royal Institution, 3. "The Greek Theatre according to Recent Discoveries," by the Rev. J. P. Mahaffy, D.D. Annual Meeting, 5.

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1953.

RESEARCHES ON THE EARTHS CONTAINED IN THE MONAZITE SANDS.

By MM. SCHÜTZENBERGER and BOUDOUARD.

CRYSTALLINE cerium sulphate obtained by the treatment of monazite sands has yielded us, on analysis, figures which lead for the corresponding metal to an atomic weight decidedly higher than that resulting from the analysis of the cerium sulphate obtained from cerite (140.5 to 141 in place of 139 to 139.5 for the formula Ce_2O_3). As this result shows the probable presence of a strange earth bordering on cerium, we have sought to isolate the latter, which we have effected in the following manner:—

1. The monazite sands, reduced to powder, are treated with sulphuric acid in heat. The excess of the sulphuric acid is evaporated, and the residue is treated with water.
2. The solution is saturated with potassium sulphate, which precipitates the earths of the cerium group in the state of double cerium sulphates, insoluble in water charged with potassium sulphates. The precipitate is washed with a saturated solution of potassium sulphate, then suspended in water, and decomposed in heat by an excess of caustic soda. The hydrated oxides, precipitated and washed, are dissolved in nitric acid and re-precipitated with ammonia, washed, re-dissolved in nitric acid, and finally precipitated by oxalic acid.
3. The oxalates, washed and dried, are converted into nitrates by nitric acid. The nitrates are dried and melted at 325° , with 8 parts of saltpetre (Debray's process), unto tranquil fusion. The cerium oxide (binoxide), insoluble, is separated from solution of saltpetre water and the nitrates of didymium and lanthanum. After washing, it is transformed into sulphate by the action of sulphuric acid; the dissolved sulphate is precipitated with oxalic acid; the washed oxalate is converted into nitrate, which is dried and melted a second time at 320° , with 8 parts of saltpetre. This second treatment serves to eliminate small quantities of didymium entangled in the first fusion. We thus obtain a light yellow oxide, finely divided, which is first converted by sulphuric acid into yellow cerium sulphate, and then into cerous sulphate, $(SO_4)_3Ce_2$, by a moderate ignition of cerium sulphate, $(SO_4)_2Ce$.

4. The white cerous sulphate is dissolved in water in the cold, and the solution is heated in a capsule on the water-bath. During the evaporation of the liquid, being kept between 75° and 80° , an abundant crystallisation separates out. When the deposit of crystals formed in heat ceases to appear, the mother-liquor is decanted off.

The crystals separated are dehydrated, dissolved in cold water, and the solution is again concentrated by heat, with separation of crystals and of a mother-liquor.

The same operation is repeated several times, and the mother-liquors obtained from the successive crystallisations are added to the first.

We thus obtain (1) a sulphate crystallised in brilliant colourless prisms, containing 13.5 per cent of crystalline water, entirely eliminated at about 300° , which we designate as A; (2), a mother-liquor, B.

The crystals A, dehydrated and dissolved in water, were precipitated with a large excess of a solution of neutral ammonium oxalate. The liquor heated for some hours on the water-bath is filtered when quite cold, and the cerium oxalate is washed with a solution of ammonium oxalate until the filtrate is no longer rendered turbid on the addition of nitric acid. The operation seems to

eliminate the traces of thorium which may have become mixed with the cerium.

The cerium oxalate is afresh converted into sulphate. The analysis of this sulphate, after desiccation at 440° , gave—(1) for 2.7196 of sulphuric anhydride, 1.6491 of calcined rose-coloured oxide; calculating this oxide as CeO_2 we have, for the atomic weight of the metal, $Ce=140.5$; (2) for 2.6017 of anhydrous sulphate we found 3.1904 barium sulphate, whence we deduce $Ce=141.05$.

The determination of the sulphuric acid by barium chloride requires certain precautions, which have been indicated by one of us in a former paper on cerium.

The augmentation of the atomic weight of the cerium of monazite cannot, therefore, be due to the presence of thorium which would have been removed by the treatment with ammonium oxalate. Still, for greater security, we applied to the dilute solution of this sulphate the method indicated by Lecoq de Boisbaudran, by heating it gently for a long time with an excess of copper oxide precipitated and dehydrated at 100° . The cupric oxide precipitates the thorium, but not the cerium. Very little copper passed into solution, and the filtrate had a greenish colour. When freed from copper by hydrogen sulphide, and concentrated on the water-bath, this liquid yields crystals, like the former in appearance, but differing in composition:—

1. Hydrated sulphate, 3.9288; anhydrous sulphate obtained, 3.4012; water, per cent, 3.47.

2. Anhydrous sulphate employed, 2.3075; oxide (white) obtained by calcination, 3.928, whence $Ce=138.6$, considering the oxide as dioxide.

3. Anhydrous sulphate employed, 2.3667; barium sulphate obtained, 2.9320, whence $Ce=138.1$.

4. Anhydrous sulphate employed, 2.7475; barium sulphate obtained, 3.4045, whence $Ce=138.1$.

5. Anhydrous sulphate employed, 2.4176; barium sulphate obtained, 2.2940, whence $Ce=137.7$.

In all the analyses effected with similar products, purified with copper oxide, the determination by calcination of the sulphate gives for the atomic weight of the cerium a value a little higher than that resulting from a determination of the sulphuric acid.

This is the inverse of that always observed on analysing cerium sulphates which have not undergone the purification with copper oxide. The atomic weight deduced from the calcination of the sulphate, and calculating the residual oxide as dioxide, is sensibly below that deduced from the contamination of the sulphuric acid.

Examination of the Mother-liquors, B.—These mother-liquors, diluted with water, are directly treated with an excess of copper oxide precipitated and dehydrated at 100° whilst in the water.

A notable quantity of copper is dissolved, and the excess of this oxide is found mixed with a large proportion of a precipitated white hydrate. The deposit is washed, and added to the precipitate formed by the A crystals under the influence of cupric oxide.

The filtrate, freed from copper by means of hydrogen sulphide, yielded crystals of cerous sulphate similar to the foregoing, that is to say to about $Ce=138.0$.

Examination of the Precipitate yielded by Copper Oxide.

—This is stirred up in water and dissolved in a slight excess of sulphuric acid. The lukewarm solution is freed from copper by means of hydrogen sulphide. It still presents very distinctly the characters of the cerium salts. On treatment with soda it gives, after the addition of an oxygenated water, an orange-yellow precipitate. On the other hand, it reveals the presence of large proportions of thorium or of analogous bodies; it is thus that, when concentrated on the water-bath, it yields an abundant white flocculent deposit formed of felted needles, which, on analysis, lead to atomic weights bordering on that of thorium.

In order to separate the cerium from the thorium, we saturated the liquid with sodium sulphate. After some time there is formed a scanty precipitate of double sulphate, which was separated after twelve hours, and

washed with a solution of sodium sulphate saturated in the cold.

This double sulphate, decomposed in heat with caustic soda, gave an oxide which when converted into sulphate offers the characters of cerium sulphate. Its analysis gave:—

1. Anhydrous sulphate employed, 2.3014; barium sulphate obtained, 2.7570, whence $Ce = 147.8$.

2. Anhydrous sulphate employed, 3.3074; calcined oxide obtained, 2.0074, whence $Ce = 141.6$.

3. Hydrated sulphate, 3.6582; anhydrous sulphate obtained, 3.1611; water per cent, 13.5.

4. Anhydrous sulphate employed, 1.7673; calcined oxide obtained, 1.0730, whence $Ce = 141.1$.

The solution of this sulphate crystallises during the evaporation in crystalline crusts, adhering to the bottom of the capsule, and which appear homogeneous to the desiccation of the last drop of the liquid. From these results we may calculate that the oxide obtained by the calcination of the sulphate has a composition close upon $2Ce_2O_3 \cdot Ce_2O_4$.

Examination of the Saturated Solution of Sodium Sulphate separated from the foregoing Precipitate.—The earth withdrawn from this liquid by means of caustic soda, washed, dissolved in nitric acid, precipitated anew with ammonia, and washed, still shows the presence of cerium compounds. Its solutions give with soda a white precipitate, which becomes reddish yellow on the addition of oxygenated water.

We succeed in separating the ceric part which occasions these coloured reactions, by treating the neutral solution of the sulphate in the cold with an excess of neutral ammonium sulphate. The greater part of the precipitate dissolves; the insoluble portion, when filtered and washed, calcined, and again transformed into sulphate, gives a salt very soluble in water and does not deposit crystals during evaporation. The liquid thickens on concentration, forming on the surface films like those of a solution of gum. On evaporation we obtain a colourless mass, amorphous and transparent, which if kept for some time in a stove at 100° becomes opaque and crystalline.

The analysis of the crystals which give the coloured reactions of cerium furnished the following numbers:—

1. Anhydrous sulphate employed, 2.566; barium sulphate obtained, 2.975, whence $Ce = 157.45$.

2. Anhydrous sulphate employed, 2.6684; calcined oxide obtained, 1.6320, whence $Ce = 144.3$, calculating the calcined oxide as bioxide. From these results we may attribute to this oxide the formula already found, that of an intermediate oxide.

The earths bordering on cerium oxide, with a high molecular weight, give, on the calcination of their sulphate, not a bioxide, but an intermediate oxide.

This explains the disagreement constantly observed between the results of the determination of the sulphuric acid, and on the calcination of the sulphate whenever it has not been purified with copper oxide.

In fine, we have separated:—

1. A cerium with an atomic weight near 138, and rather lower, the solution of which does not precipitate copper oxide.

2. A cerium of an atomic weight near 148, the sulphate of which is precipitable by copper oxide, and also by sodium sulphate.

3. A cerium of an atomic weight near 157, the sulphate of which is precipitable by copper oxide, but not by sodium sulphate. The solutions are characterised by taking a gummy aspect during concentration.

These three earths give yellow ceric salts, decomposable by heat into white cerium salts. Oxygenated water with soda precipitates them of an orange-red. Ammonium oxalate precipitates them, all three, and the precipitates are not soluble in excess. Spectroscopic examination (sparks with chloride) does not show any difference between the three salts.

The part soluble in neutral ammonium oxalate, and not

precipitated by sodium sulphate, does not present the coloured reactions of cerium, but belongs to the thorium group. The experiments hitherto effected have led us to a homogeneous product.—*Comptes Rendus*, cxxiv., p. 481.

THE BEHAVIOUR OF BACTERIA WITH CHEMICAL REAGENTS.

By TH. PAUL and B. KRÖNIG.

FROM the authors' experiments with the spores of splenic fever and those of *Staphylococcus pyogenes* it results that—

1. With the exception of platinum, the salts of gold, silver, and mercury have a specific toxic action.

2. The disinfective action of the metallic salts depends not alone on the concentration of the metal in solution, but also on the specific properties of the salts and the solvent. Behring's view that the disinfective value of the mercury solutions depends alone on the percentage of soluble mercury cannot be rightly admitted.

3. Solutions of metallic salts in which the metal is an ingredient of a complex ion, and the concentration of its ion is hence very trifling, exert merely a very slight antiseptic action.

4. The effect of a metallic salt depends not merely on the specific action of the metallic ion, but also on that of the anion.

5. The haloid compounds of Hg (including the cyanides and sulphocyanides) disinfect according to their degree of dissociation.

6. The disinfective action of aqueous mercuric chloride is lowered by the addition of metallic chlorides.

7. The strong acids act at concentrations of 1 litre and upwards, not only corresponding to the concentration of their hydrogen ions, but also by means of the specific properties of the anion. The strong acids, even if more dilute, and the weak organic acids seem to act in the proportion of their degree of dissociation.

8. The bases KOH, NaOH, and LiOH, when dissociated, disinfect almost equally; $NH_4(OH)$, when much less dissociated, disinfects very little.

9. The oxidising agents, NH_3 , $H_2Cr_2O_7$, $HClO_3$, $HMnO_4$, act according to their position in the series laid down according to their electric behaviour. Chlorine does not rank in this series, but has a very powerful specific action.

10. The disinfective action of the halogens, Cl, Br, I, decreases, like their general chemical behaviour, with the increase of atomic weight.

11. The statements Scheurlein that solutions of phenol disinfect better on the addition of salts was verified.

12. The known fact that substances dissolved in alcohol and ether are almost without action on the spores of splenic fever was confirmed by the author's observations.

13. Aqueous alcohol of known percentage heightens the disinfective action of $HgCl_2$ and $AgNO_3$.—*Chemiker Zeitung*.

Determination of Organic Matter in Potable Water.
—Dr. E. Fricke.—It is an unpleasant property of centinormal oxalic acid that it gradually loses its efficiency. A recent solution of oxalic acid of which 10 c.c. are just faintly reddened by an equal volume of permanganate, after being kept for eight days required only 9.5 c.c. for adjustment, and after four weeks its value had fallen to 8 c.c. permanganate. The appearance of flocculent matter showed that the oxalic acid is decomposed by fungi. As it is very inconvenient to prepare a new standard solution every day or two I tried adding to the oxalic solution 1 grm. boric acid per litre, and I find that after ten weeks it has remained unchanged. The boric acid has no action upon the permanganate.—*Chem. Zeitung*, No. 26, 1897.

THE IRONSTONE OF THE WEALD.

By Dr. T. L. PHIPSON.

Of the various ironstones examined of late years in this laboratory, none are more interesting than that of the Weald. This particular portion of the Earth's crust, which lies just below the green sand of the chalk formations, is rarely met with on the surface of the globe. It presents the characters of a fresh-water formation, and has been found to contain the remains of some gigantic reptiles. There exist a few small patches of it in the South of England. The fresh-water *Paludina* shells are one of its characteristics.

I have made a careful examination of the ironstone of this formation, and have found that it consists of a yellowish white or grey carbonate of iron, generally coated with hydrated peroxide in the form of brown hæmatite, which in its turn passes into reddish orange or yellow ochraceous stones containing much silica.

The carbonate of iron of the coal measures is also, evidently, of fresh-water origin; for, among the ores of this kind forwarded to me from South Staffordshire, I have met with a beautiful little specimen of a fresh-water mollusc (*Unio*) presenting exactly the composition of these spathic ironstones.

But whilst the carbonate of iron of the coal strata contains a notable amount of phosphates and sulphates, that of the Weald is remarkably pure in this respect.

The Weald ironstone has given me from 34 to 42 per cent of metallic iron; 14 to 46 per cent of silica (which is the chief impurity), and about 2 to 3 per cent of oxide of manganese, with similar quantities of magnesia and alumina, and a little lime, but only faint traces of sulphates and phosphates.

These spathic ironstones seem to have been formed from ancient chalybeate springs, such as are rather frequent at the present day, especially in Germany, where the process is still in action, the iron being dissolved from the rocks through which issue constantly vast streams of carbonic acid. Near Neubau, in Waldeck, for instance, I have seen bubbles of carbonic acid varying in size from that of a walnut to that of a man's head, bursting on the surface of springs, incessantly, day and night; and this volcanic action has been continuous there as long as man can remember.

The almost complete absence of phosphorus and sulphur in the ironstone of the Weald is rather remarkable, since spathic ironstone is generally different in this respect. In olden times, when forests abounded in the South of England, this ore was smelted and made good iron. I have heard it stated that the old iron-railings of St. Paul's Cathedral were obtained from this source; and among the samples forwarded to me for analysis, and picked up on the surface of the ground, I have met with pieces of slag that appear to be Catalan slag.

Casa Mia Laboratory, Putney, S.W.,
April 27, 1897.

MODIFICATION OF THE THALLEOQUIN TEST FOR QUININE.

By F. S. HYDE.

It is extremely important for the success of this test that the reagents employed should be dilute. Some authorities give the quantity of each reagent necessary, without stating the proper dilution, thereby causing much inconvenience.

The light green colouration produced on porcelain by contact of the quinine salt with weak bromine or chlorine water and ammonia, is not nearly so striking as the brilliant emerald-green colour obtained by using dilute solutions in a test-tube.

Usually the analyst deals with unknown quantities, or mere traces, but for experiment it will be found convenient to use from 3 to 5 m.grms. of the quinine salt for each test. (With larger amounts there is a tendency to form bulky precipitates).

For example, place 3 to 5 m.grms. (0.003 to 0.005 grm.) quinine sulphate in a test-tube, and add about 5 c.c. distilled water. Acidulate with one drop (not more) of dilute sulphuric acid (1:4), which immediately dissolves the quinine sulphate with a blue fluorescence. An excess of the acid should be avoided.

At this point various authorities recommend the addition of weak bromine or chlorine water; but the writer has found that if a clear, filtered solution of calcium hypochlorite (bleaching powder) be substituted for the bromine or chlorine water, the results will be more satisfactory so far as certainty and brilliancy of the test are concerned.

The points to be observed are as follows:—After acidulation with one drop of sulphuric acid (1:4) the hypochlorite solution is added through a small filter to the quinine solution in the test-tube, until the blue fluorescence just disappears, and the solution acquires a faint golden tint; then add a few drops of dilute ammonia (1:3), when a clear emerald-green colour should appear. (Thalleoquin test).

The tint thus produced seems to be more brilliant than that obtained through the agency of bromine water.

On the addition of a slight excess of dilute sulphuric acid to this green solution, a blood-red tint will be produced, which may be considered confirmatory. This is not always the case, however, when bromine water has been used in the preliminary operation.

Potassium or sodium hypobromite is not applicable, on account of the strong alkali, which tends to precipitate the white quinine base, and thus interfere with the brilliancy of the test. Chlorinated soda (Labarraque's solution) likewise gives uncertain results, the tints varying from yellowish green to violet.—*Journal of the American Chemical Society*, xix., p. 331.

THE ACTION OF ACID VAPOURS ON METALLIC SULPHIDES.*

By JEROME KELLEY, Jun., and EDGAR F. SMITH.

EXPERIMENTS made in this laboratory on the action of the vapours of hydrochloric acid upon the sulphide of arsenic proved that the latter is wholly volatilised. The purpose of the present communication is to record further observations along analogous lines. Thus, when washed and dried arsenic trisulphide is exposed to the action of hydrobromic acid gas, it volatilises completely. Indeed, the action commences in the cold with the formation of a liquid that passes out of the containing vessel upon the application of a very gentle heat. In evidence of this, two quantitative experiments may be given:—

Arsenic sulphide taken. Grm.	Arsenic sulphide expelled. Grm.
0.2945	0.2941
0.4632	0.4628

Antimony trisulphide, like that of arsenic, is volatilised by hydrochloric acid gas. It was quite probable that a like deportment would be observed if hydrobromic acid gas should be substituted. This was found to be the case. When the gas came in contact with the sulphide it became liquid and volatilised as soon as a gentle heat was played upon the boat in which the sulphide was contained.

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xviii., No. 12.

Antimony sulphide taken. Grm.	Antimony sulphide expelled. Grm.
0.1473	0.1469
0.0938	0.0935

Upon substituting stannic sulphide for antimony sulphide, an experience similar to that observed with antimony and arsenic sulphides followed. There was a complete volatilisation with but a trifling residue, which proved to be carbon from filter-paper that had adhered to the metallic sulphide.

Stannic sulphide taken. Grm.	Stannic sulphide expelled. Grm.
0.1880	0.1880
0.5527	0.5521
0.4174	0.4169

The oxides of arsenic, antimony, and tin (at least in the stannic form) can be volatilised in a current of hydrochloric acid gas. This is also true of the sulphides of arsenic and antimony, but how the two sulphides of tin would act under like conditions was not known.

Experiments recently made demonstrate the perfect volatility of stannic sulphide in this way. With stannous sulphide it was found that by the continued action of the gas in the cold there followed a complete conversion into chloride without any volatilisation. That the residue was the chloride was evident from its action upon a mercuric salt solution. The figures obtained in the several trials were:—

Stannous chloride found. Grm.	Stannous chloride theory. Grm.
0.3544	0.3523
0.4893	0.4903

Several attempts were made to separate stannous and stannic sulphides by this procedure. The results were unsatisfactory. In order to drive out the stannic salt completely it is necessary to heat the mixture, and this caused a partial volatilisation of the stannous chloride, so that quantitative results could not be obtained.

Comparatively few metallic sulphides have been studied in the direction indicated in the preceding lines, so that it is probable that a wider application of the method will disclose interesting behaviours, and that probably new separations can be brought about in this way. The action of the vapours of haloid acids has also been tried on natural sulphides with a fair degree of success.

THE ESTIMATION OF MOLYBDENUM IODOMETRICALLY.*

By F. A. GOOCH.

IN a former paper from this laboratory (Gooch and Fairbanks, *Am. Journ. Sci.*, IV., ii., 157, 1896) several modes of applying hydriodic acid to the reduction of molybdic acid were studied. It was found, first, that the digestion process of Mauro and Danesi (*Zeit. Anal. Chem.*, xx., 507) is of very limited applicability, owing to the fact that the reaction of reduction is reversible. Secondly, it appeared that the use of the same reaction by Friedheim and Euler (*Ber. d. D. Chem. Gesell.*, xxviii., 2066) in a distillation process, so arranged that the iodine set free in the reduction might be caught in the distillate and titrated to serve as the measure of the reducing action, was not sufficiently regular because of inattention to minor details. It was shown that by taking care to adjust the conditions constant results might be obtained.

Thirdly, the fact was developed that by simply boiling the solution under well-defined conditions in an ordinary Erlenmeyer flask, partly closed by a simple trap, the reduction of the molybdic acid proceeded regularly, and that the addition of standard iodine to the solution made alkaline with sodium bicarbonate served to restore the original condition of oxidation of the molybdic acid. The results of this treatment were shown to be accurate.

In a recent paper (*Ber. d. D. Chem. Gesell.*, xxix., 2981) Friedheim has seen fit to make our modifications of the distillation process the subject of attack. Friedheim's comments upon the third method discussed—as well as upon a subsequent application of the process ("An Iodometric Method for the Determination of Phosphorus in Iron," by Charlotte Fairbanks)—are evidently prompted wholly by personal opinion and demand no further attention. With reference to Friedheim's denial of the necessity of modification in the Friedheim and Euler treatment the case is different.

The process of Friedheim and Euler consists, it will be remembered, in treating the soluble molybdate, or the solution of molybdic acid in sodium hydroxide, with potassium iodide and hydrochloric acid in a Bunsen apparatus, boiling until the solution is of a clear green colour, collecting the iodine distilled in potassium iodide, and titrating it with sodium thiosulphate. We found that the development of the green colour was not a sufficient criterion of the exact reduction of the molybdic acid to the condition of the pentoxide and of the removal of the iodine which should be theoretically set free. To accomplish that end we found it safer and more convenient to start the distillation with a definite volume (40 c.m.³) of liquid and boil until a definite volume (25 c.m.³) was reached, care being taken with regard to the strength of acid and the excess of potassium iodide employed. Experience showed unmistakably that in order to avoid the decomposing action of the air upon the hot vaporous hydriodic acid in the retort, it was necessary to go beyond the measures advised by Friedheim and Euler (namely, to warm the retort and its contents slowly, heating to boiling only when the connecting tube was well filled with iodine vapour, and the tendency toward back-suction of the liquid in the receiver began to appear), and to conduct the operation in a simple little apparatus (the retort holding about 100 c.m.³) put together entirely with sealed and ground joints, as shown in the figure of the former paper, so arranged that a current of purified carbon dioxide could be passed through retort and receiver during the distillation. With this apparatus we were able to determine with accuracy the point of concentration at which the free iodine left the liquid, the molybdic acid having been converted to the condition of the pentoxide. It was found that if dependence is placed upon the occurrence of the so-called clear green colour of the liquid to determine the end of the distillation, it may frequently happen that free iodine remains in the residue. This takes place, it will be observed, in the atmosphere of carbon dioxide, so that the presence of the free iodine can by no possibility be attributed to the action of atmospheric air upon the hydriodic acid remaining after the distillation is complete. On the other hand, it appeared that, if the distillation is pushed too far, the molybdenum pentoxide may be still further reduced, with consequent evolution of more than the expected amount of iodine. The attainment of an exact degree of reduction with the expulsion of the corresponding amount of iodine becomes, therefore, a matter of chance unless further precautions are taken. We found in our experiments that if amounts less than 0.3 grm. of the molybdic acid are introduced in soluble form into the 100 c.m.³ retort with a not too great excess of potassium iodide, and the 40 c.m.³ of liquid so constituted that 20 c.m.³ of it shall be water and 20 c.m.³ the strongest hydrochloric acid, the reduction proceeds with a fair degree of regularity in the manner expected. We found it important to restrict the excess of potassium

* Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, Fourth Series, vol. iii., March, 1897.

iodide so that it shall never exceed the theoretical requirement by more than 0.5 grm.

Our determinations with the pure molybdenum trioxide showed errors varying from 0.0010 grm. + to 0.0007 grm. -; the variations from theory in the experiments with ammonium molybdate ranged from 0.0011 grm. + to 0.0011 grm. -. If these results are compared with those given by Friedheim and Euler, the advantage is a little in favour of the latter; but a scrutiny of the figures given by Friedheim and Euler develops the fact that the apparent accuracy of their work is founded upon miscalculations. This fact was known to us at the time of our former writing, but we did not consider it essential then to make the matter public. The recent attack of Friedheim makes that course now necessary.

Herewith is reproduced a table of results obtained by Friedheim and Euler in the test of their method upon ammonium molybdate, shown by analysis to contain 81.49 per cent of molybdenum trioxide. The figures which are incorrect are enclosed in brackets:—

Original Figures of Friedheim and Euler.

Molybdate taken. Grm.	Na ₂ S ₂ O ₃ used. C.m.s.		MoO ₃ found. Grm.	Per cent of MoO ₃ referred to molybdate taken.
0.2674	30.8	I c.m. ² =	0.2184	[81.71]
0.4418	50.8		0.3601	81.51
0.4075	[40.7]*	MoO ₃ .	0.3317	81.40
0.3281	37.33		0.2644	[81.85]
0.4340	49.43	I c.m. ³ =	0.3502	[81.69]
0.4098	46.63		0.3304	[81.67]
0.4305	49.08	MoO ₃ .	0.3478	[81.78]

* Probably 46.7.

Appended is a re-calculation of the percentage of the trioxide found, with columns showing the percentage error and the error stated in fractions of a grm. Changes from the figures of Friedheim and Euler are in modern-faced type.

Re-calculation of the Results of Friedheim and Euler.

Corrected per cent of MoO ₃ found, referred to the molybdate.	Error in per cent of MoO ₃ found compared with MoO ₃ taken.	Error of MoO ₃ . Grm.
81.68	0.23+	0.0005+
81.51	0.03+	0.0001+
81.40	0.12-	0.0004-
80.58	1.12-	0.0030-
80.69	0.99-	0.0035-
80.62	1.05-	0.0035-
80.79	0.86-	0.0030-

These figures of their own (properly calculated) are sufficient to show the inadequacy of the method of Friedheim and Euler. We ourselves were occasionally able to get results from the method of Friedheim and Euler quite as good as these; it must be said, however, that most of our results obtained by their unmodified method have been even worse than their own.

In another series of six determinations, in which molybdenum trioxide was the starting point, Friedheim and Euler were more successful, the errors varying from 0.0006 grm. + to 0.0006 grm. -. Thus, Friedheim and Euler establish by their own results the fact that the hitting of the right point at which to stop their process of boiling is a matter of chance. In spite of the probability that some of the iodine which they found in the receiver was liberated by atmospheric action, the fact remains that their results are in many cases very low. That is, they did not boil long enough.

The difficulty appears again in the modification of their method which Friedheim and Euler apply to the determination of molybdenum trioxide associated with vanadium pentoxide (*Ber. d. D. Chem. Gesell.*, xxviii., 2072), namely, the distillation with phosphoric acid and

potassium iodide of the residue left after reducing the vanadium pentoxide by hydrochloric acid and potassium bromide, according to the method of Holverschreit. We reproduce the part of their table which refers to the determination of the molybdenum, adding, however, columns containing the errors and corrected percentages.

MoO ₃ taken.	MoO ₃ found.	Per cent MoO ₃ F. and E.	Error. Grm.	Per cent MoO ₃ Re-calculated.
0.15037	0.15005	99.79	0.00032-	99.79
0.16895	0.16879	99.90	0.00016-	99.90
0.17758	0.17729	99.84	0.00029-	99.84
0.24975	0.24962	99.95	0.00013-	99.95
0.33151	0.33607	[99.87]	0.00456+	101.38

Four of the five determinations are accurate, but the fact that all figures are carried out to the fifth decimal place does not keep three good sized figures out of the error column for the fifth determination.

It is hardly necessary, in the light of a comparison of the results of Friedheim and Euler with ours, to discuss further the unreliability of the unmodified process. The necessity of a proper control of the volume, strength of acid and excess of potassium iodide, as well as proper protection from atmospheric oxidation, is real.

On a former occasion the unpleasant necessity presented itself (Gooch and Browning, *Am. Journ. Sci.*, xlv., 334) of pointing out the fact that certain unfounded criticisms on the part of Friedheim and Meyer were based upon an unfortunate use by them of impure reagents; the difficulty in the present case, for Friedheim and Euler, seems to reside in the arithmetical process.

DETERMINATION OF POTASH AND PHOSPHORIC ACID IN FODDERS.

By H. W. WILEY.

In the comparative analyses of soils during the past three years we have grown a great number of pot cultures and determined the mineral plant-foods in the resulting crops. The following modified potash method, devised by Mr. K. P. McElroy, while not sacrificing accuracy, has made it possible for one analyst to determine the potash, often in duplicate, in more than ten samples a day. Since the quantity of the crop harvested from a poor soil is often small, it is desirable that the phosphoric acid and potash be determined in the same sample.

The method in use for the determination of potash in feeding stuffs, in the laboratory of the United States Department of Agriculture is a simple modification of the ordinary Lindo-Gladding method, as prescribed by the Association of Official Agricultural Chemists. It is as follows:—

1. Burn 8 grms. of the substance over a low flame to a proximate whiteness. Burning after addition of sulphuric acid does not give more potash than burning alone, and it is more troublesome. Transfer the ash to a 200 c.c. flask, using about 50 c.c. of water; add 5 c.c. of strong hydrochloric acid, and place on the steam-bath for an hour, or boil from five to ten minutes. Add a little iron chloride to precipitate all phosphoric acid as ferric phosphate, then 10 c.c. of strong ammonia, and then from 5 to 10 c.c. of ammonium carbonate solution (200 grms. per litre of the commercial salt). Replace on the steam-bath and heat for an hour, and allow to stand over night. Complete the volume to the 200 c.c. mark with water, and shake three times at intervals of five or ten minutes. Grease the inside of the neck of the flask, and pour its contents on a dry folded filter. When all is transferred to the filter and run through, wash down the neck of the flask with a little water, put the funnel into the flask, and stand aside till the filter dries. The roll up the filter, and push down

into the flask. Add dilute nitric acid, digest, make volume up to the mark, and use an aliquot part for the determination of phosphoric acid.

Transfer 50 c.c. of the filtrate containing the potash, equivalent to 2 grms. of material, to a platinum dish, cover, and heat on the steam-bath till evolution of gas ceases. Remove the cover, and rinse it and the sides of the dish with a stream from the wash-bottle. Evaporate to dryness, and heat in an air-bath till all water is removed in order to avoid loss by decrepitation in the subsequent ignition. Heat over a low gas flame till the bulk of the ammonium chloride is removed, cool, and add 1 c.c. of sulphuric acid (1 : 1); then heat on a hot plate till fuming begins, then over a flame till all the sulphuric acid is driven off and the residue in the dish is white. Every portion of the dish should reach a low but distinct red heat, the bottom first and then the sides. The reason for the preliminary driving off of the bulk of the ammonia as sal-ammoniac is that ammonium sulphate melts and sputters, involving danger of loss. Cool the dish, and add one or two drops of strong hydrochloric acid, then from 50 to 75 c.c. of water, washing down the sides of the dish with a jet from the wash-bottle. Add platinum chloride solution in amount equivalent to 150 m.m. of metallic platinum for materials not containing over 4 per cent of potash. Very few reach this limit. Evaporate on the water-bath as usual, and take up with alcohol of 80 (volume) per cent. Filter through a Gooch crucible, keeping the insoluble material in the dish as far as possible. Wash with four more portions of alcohol, decanting through the crucible each time. Finally rinse down the sides of the crucible with a stream of alcohol from a wash-bottle. Cover the residue in the dish with the half-saturated solution of ammonium chloride prescribed in the official method for the determination of potash, and stir thoroughly. Decant through the Gooch crucible, and treat with five or more portions of sal-ammoniac solution, decanting through the crucible each time. Finally wash into the crucible with 80 per cent alcohol. When the transfer is complete, rinse the sides of the crucible thoroughly, and finally fill it twice with alcohol, of course constantly filtering with a vacuum. Dry for an hour at 100° and weigh.

Pour about 150 c.c. of boiling water through the weighed Gooch crucible. If the platinum potassium chloride is not wholly dissolved, again bring the filtrate to a boil and pour through once more. Store this filtrate finally in a large flask, containing aluminium clippings, to reduce the platinum. Bring a fresh portion of water (150 c.c.) to a boil, and pour through the Gooch crucible. Remove the crucible from the vacuum apparatus, wipe, and dry in an air-bath, with good ventilation, for two hours, at 110°. Weigh once more. The loss in weight is the double chloride. The second portion of hot water is used to dissolve the double salt in the next crucible operated upon, after being once more brought to a boil. —*Journal of the American Chemical Society*, xix., p. 320.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Extra Meeting, March 25th, 1897.

Mr. A. G. VERNON HARCOT, President, in the Chair.

PROFESSOR P. FRANKLAND, Ph.D., B.Sc., F.R.S., delivered the Pasteur Memorial Lecture.

It was pointed out that, although the name of Pasteur was associated with the progress of so many branches of science, it appeared that his remarkable success in such varied fields of investigation was in large measure due to the chemical discipline under which he had grown up,

and in his having almost for the first time extended the experimental methods and close reasoning of the chemist to other sciences, in which previous investigators had been mostly occupied with matters of a superficial character. Reference was then made to Pasteur's birth, on December 27, 1822, his early life and entrance as a student into the École Normale, to his being retained as an assistant there by Balard, and to his interest being awakened in the study of crystallography by Delafosse. His classical researches on the tartrates and malates were then described, and the principal results discussed from a practical and theoretical point of view. It was shown of what enormous importance for the development of chemical theory had been the introduction by Pasteur of the conception of molecular symmetry and dissymmetry, whilst the wide applicability of his methods of investigating stereoisomeric compounds was indicated in outline. The manner in which he was next led to investigate fermentation phenomena was then described, his conflict with Liebig was touched upon, and his overthrow of the so-called "chemical fermentation theory" detailed. Reference was then made to his participation in the spontaneous generation controversy, and to his victories over Pouchet, Joly, Musset, and Bastian, the last champions of the doctrine of abiogenesis. The practical aspects of Pasteur's fermentation studies were briefly considered, the famous "Études sur la bière"; "sur le vin," and "sur la vinaigre," as well as process known as *pasteurisation*. The direct influence of these fermentation studies on medicine, surgery, and public health was pointed out, and the manner in which they had led to the magnificent work of Lister on the antiseptic treatment of wounds was indicated. Pasteur's investigation of the destructive silkworm diseases, *pébrine* and *flacherie*, was then dealt with, and his gradual attraction into the field of pathological research traced. His studies on anthrax, his recognition of the bacillus of malignant oedema, and his discovery of the *Staphylococcus pyogenes* and of the *Streptococcus pyogenes* were referred to. Then the manner in which he came upon the possibility of attenuating viruses and preparing vaccines were described; the lecture concluded with an account of the methods of protective inoculation devised by Pasteur, first for fowl-cholera, then for anthrax, for swine-measles, and his final triumph in successfully elaborating a curative treatment for rabies.

LORD LISTER, P.R.S., proposed, and Sir HENRY ROSCOE seconded, a vote of thanks to Professor PERCY FRANKLAND, which was carried unanimously.

Anniversary Meeting, March 31st, 1897.

Mr. A. G. VERNON HARCOT, President, in the Chair.

Professor Collie, F.R.S., and Dr. Wynne, F.R.S., were appointed scrutators, and a ballot was opened for the election of Officers and Council for the ensuing year, the ballot being closed at the conclusion of the President's Address.

The Longstaff Medal was then presented to Professor Ramsay for the discovery of helium, and for his share in the investigation of argon. The President referred to the circumstances under which the first announcement of the discovery was made, and expressed the pleasure he felt in presenting him with the Medal.

The President began his Address by thanking the Fellows, and more especially the Officers and Council, for the kindness with which they had aided him during his year of office, and expressed his pleasure at the progress of the Society during the past year. He referred to the arrangements made for the reading of papers, and suggested means by which the discussions might be made more useful.

The publication of the Jubilee volume, largely drawn

up by Dr. Armstrong, was mentioned, and the services rendered by Mr. Warington in its preparation were acknowledged. The Hofmann Memorial Lectures had also been issued. Lothar Meyer and Pasteur Memorial Lectures had been delivered in the year, and arrangements had been made for the Kekulé Lecture. Reference was also made to the work of the Council through the year, to the death of Sir William Grove, an original member of the Society, and to the munificent donation of one thousand guineas by Mr. J. J. Tustin.

The remainder of the Address was devoted to the consideration of the question whether the changes which matter undergoes are different in their nature.

The numerical strength of the Society was as follows:—

Number of Fellows, March 31st, 1896	2019
since elected	130
reinstated by Council	7
	2156
Removed on account of non-payment of two	
annual subscriptions	25
Withdrawn	29
Deaths	23
	77
Number of Fellows, March 31st, 1897	2079
Foreign Members	27

The following have died:—I. M. T. Anderson; John Curragh; Captain Marshall Hall; G. Harley, F.R.S.; James Hart; John Hughes; W. Lapraik, Ph.D.; J. B. L. Mackay; A. H. Mason; H. A. Nott; Baron F. von Mueller, F.R.S.; W. J. Palmer; Sir J. Prestwich, F.R.S.; Edward Rawlins; G. F. Schacht; James Scorgie; T. Shimidzu; T. J. Smith; Charles Tomlinson, F.R.S.; W. H. Walenn; Richard Weaver; W. H. Wood; T. G. Wormley, M.D.

The number of communications made to the Society during the year was 173.

One hundred and seventeen papers and three Memorial Lectures were published in the *Transactions* for 1896, occupying 1702 pages, whereas in the preceding year 116 papers were published, occupying 1172 pages.

The following were the statistics relating to the Abstracts:—

PART I.		
	Pages.	No. of Abstracts.
Organic Chemistry	716	1201
PART II.		
General and Physical Chemistry		319
Inorganic Chemistry		287
Mineralogical Chemistry		267
Physiological Chemistry		182
Chemistry of Vegetable Physiology and Agriculture		153
Analytical Chemistry		430
Total in Part II.	684	1638
Total in Parts I. and II.	1400	2839

Eight hundred and fifteen volumes had been borrowed from, and 163 books, 310 volumes of periodicals, and 24 pamphlets added to the Library.

Professor ODLING, F.R.S., proposed a vote of thanks to the President, coupled with the request that he would allow his Address to be printed in the *Transactions*.

Dr. FRANKLAND, F.R.S., seconded the motion, which was carried by acclamation.

The PRESIDENT having returned thanks,

Dr. THORPE, F.R.S., the treasurer, gave an account of the balance sheet, which he laid before the Society, duly audited.

The receipts had been:—By admission fees and subscriptions, £4134; by sale of Journal and advertisements, £702 14s. 6d.; and by dividends on invested capital, £475 10s. 6d. The expenses had been:—On account of the Journal, £3089 8s. 1d.; on account of the Proceedings, £237 18s. 1d.; on account of the General Index, £464 3s. 9d.; on account of the Library, £355 2s. 0d.; on account of the Jubilee, £336 12s. 1d.; House expenses, £199 17s. 4d.; the total expenditure being £5385 4s. 7d. Grants amounting to £308 had been made to the Fellows from the Research Fund during the year.

Sir F. ABEL, F.R.S., proposed that the thanks of the Fellows be tendered to the Treasurer for his services during the past year; this motion was seconded by Mr. PHIPSON BEALE, Q.C., and carried.

The TREASURER, in responding, proposed a vote of thanks to the auditors.

Mr. J. H. M. PAGE seconded the motion, which was unanimously adopted, and acknowledged by Mr. H. B. BAKER.

Dr. W. J. RUSSELL, F.R.S., proposed a vote of thanks to the Officers and Council.

Professor TILDEN, F.R.S., seconded the motion, which was unanimously adopted.

Dr. DYER responded on behalf of the Council.

Professor H. B. DIXON, F.R.S., proposed a vote of thanks to the Editor, Sub-Editor, Abstractors, and Indexers, which was seconded by Mr. FRISWELL, and carried.

Mr. GROVES, F.R.S., responded.

The scrutators having presented their report to the President, he declared that the following had been duly elected:—

President—James Dewar, M.A., LL.D., F.R.S.

Vice-Presidents who have filled the office of President—

Sir F. A. Abel, Bart., K.C.B., D.C.L., F.R.S.; H. E. Armstrong, Ph.D., LL.D., F.R.S.; A. Crum Brown, D.Sc., M.D., F.R.S.; W. Crookes, F.R.S.; E. Frankland, D.C.L., F.R.S.; Sir J. H. Gilbert, Ph.D., F.R.S.; J. H. Gladstone, Ph.D., F.R.S.; A. Vernon Harcourt, D.C.L., F.R.S.; H. Müller, Ph.D., F.R.S.; W. Odling, M.B., F.R.S.; W. H. Perkin, LL.D., Ph.D., F.R.S.; Lord Playfair, Ph.D., K.C.B., F.R.S.; Sir H. E. Roscoe, LL.D., F.R.S.; W. J. Russell, Ph.D., F.R.S.; A. W. Williamson, LL.D., F.R.S.

Vice-Presidents—Francis Robert Japp, M.A., Ph.D., LL.D., F.R.S.; Ludwig Mond, F.R.S.; William Ramsay, Ph.D., F.R.S.; J. Emerson Reynolds, D.Sc., F.R.S.; W. Chandler Roberts-Austen, C.B., F.R.S.; William A. Tilden, F.R.S.

Secretaries—J. Millar Thomson; Wyndham R. Dunstan, M.A., F.R.S.

Foreign Secretary—Raphael Meldola, F.R.S.

Treasurer—T. E. Thorpe, LL.D., F.R.S.

Other Members of Council—P. Phillips Bedson, D.Sc.; Bennet Hooper Brough; Otto Hehner; C. T. Heycock, M.A., F.R.S.; Herbert McLeod, F.R.S.; Rudolph Messel, Ph.D.; H. Forster Morley, M.A.; James Wyllie Rodger; T. Kirke Rose, D.Sc.; Alexander Scott, M.A., D.Sc.; Arthur Smithells, B.Sc.; Sydney Young, D.Sc., F.R.S.

The question having been raised as to whether the number of votes cast for each candidate for the Presidency should be declared, the PRESIDENT stated that this had not been the custom, but he would take the sense of the meeting on the point. A majority being in favour of a declaration of the numbers, the President conferred with the scrutators and then stated that there was a difficulty in announcing the numbers, owing to a question having arisen in reference to the validity of certain voting papers, in which the instruction to erase the printed name had not been complied with.

Inasmuch as the rejection of these irregular papers would only increase the majority and not affect the result

of the election, and as it now appeared that the announcement of the numbers would involve re-counting the votes, the President suggested that the Fellows should be content with the scrutators' report. The ruling of the President as to the validity of the irregular papers having been requested, he stated that, in his opinion, they were invalid.

Questions having been asked as to the by-laws governing the election, the PRESIDENT stated that the election had been conducted in strict accordance with the by-laws, and he therefore declared the election valid.

In the evening, at 7 p.m., the Fellows and their friends dined together at the Criterion Restaurant, Mr. A. G. Vernon Harcourt, the retiring President, in the Chair.

The following toasts were proposed:—

By the CHAIRMAN: 1. Her Most Gracious Majesty the Queen. 2. Their Royal Highnesses the Prince and Princess of Wales and the others members of the Royal Family.

By the Right Hon. Lord LISTER, President of the Royal Society: 3. Prosperity to the Chemical Society.

By WILLIAM CROOKES, Esq., F.R.S.: 4. The Learned and Scientific Societies, coupled with the name of Sir John Evans, K.C.B., Treasurer of the Royal Society.

By Dr. J. H. GLADSTONE, F.R.S.: 5. The Guests, coupled with the name of Professor Michael Foster, Secretary of the Royal Society.

By Dr. W. J. RUSSELL, F.R.S.: 6. The retiring President.

By Dr. ARMSTRONG, F.R.S.: 7. The Secretaries, coupled with the name of Professor J. M. Thomson.

Ordinary Meeting, April 1st, 1897.

Prof. DEWAR, F.R.S., President, in the Chair.

Messrs. William Douglas, Ernest Goulding, T. H. Lee, and W. A. Davis were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. William Barlow, Hillfield, Muswell Hill, N.; James Brierley, 12, Brunswick Square, Southampton; Alexander Duckham, Crooms Hill, Greenwich Park, S.E.; Harold William Harrie, 298, Amhurst Road, Stoke Newington, N.; Sydney Hill, 11, Salisbury Street, Hull; Willie Lee Mallinson, Gawthorp Green, Kirkheaton; Edmund Howd Miller, M.A., Ph.D., Columbia University, U.S.A.; Joseph Previte Kennedy Orton, B.A., Ph.D., 20, Loughborough Road, Brixton, S.W.; Charles Alfred West, 105, Sydney Street, Chelsea, S.W.; Paul Thomas White, Horton Field, West Drayton.

Mr. CASSAL asked whether the President would take steps to carry into effect the wishes of a majority at the Anniversary Meeting, that a recount of the ballot papers should be made, and the votes recorded for the two nominees for the Presidency announced to the Fellows.

Mr. VERNON HARCOURT said that, although a majority at the Anniversary Meeting had declared themselves in favour of the announcement of the numbers, it had not been found possible to make any exact announcement without going through the voting papers again, and he had hoped that, having regard to all the circumstances, it would be generally felt best to accept the result of the election as it had been recorded by the Scrutators, especially as a succinct and accurate account of what had happened at the Anniversary Meeting had been included in the minutes.

The PRESIDENT said he considered the subject closed, but promised to bring the question before the Council. He could not see that any good would result from a recount of the voting papers for the mere purpose of delaring the exact numerical majority by which the

President had been elected. He hoped that long before the Council met the matter would be forgotten.

Of the following papers those marked * were read:—

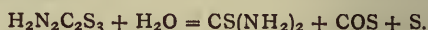
*50. "*The Hydrolysis of Perthiocyanic Acid.*" By F. D. CHATTAWAY, M.A., and H. P. STEVENS, B.A.

When potassium thiocyanate is treated with sulphuric acid, many different substances are produced; thiocyanic acid, however, is always first liberated, and then reacts in various ways determined by the conditions of the experiment.

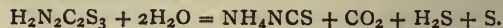
The best known reaction, usually represented as a simple hydrolysis of thiocyanic acid, is that by which carbon oxysulphide is commonly prepared. Other actions, however, go on, and the carbon oxysulphide is invariably mixed with carbon dioxide, sulphur dioxide, hydrocyanic acid, and carbon bisulphide.

The authors have observed that, in addition to these, a considerable amount of thiourea is produced. This thiourea has been found to be a decomposition product of perthiocyanic acid, which is always formed in considerable quantity when acids act upon thiocyanates, and the paper deals mainly with the hydrolysis of this acid.

Perthiocyanic acid is easily hydrolysed, either by heating with water under pressure, or by heating with strong sulphuric acid, thiourea, carbon oxysulphide, and sulphur being formed.—



As the action only takes place at a comparatively high temperature, one or other of these products is invariably decomposed. When perthiocyanic acid is heated with water to about 200° in closed tubes, this action may be considered to take place first; but at the high temperature the thiourea is transformed completely into ammonium thiocyanate, while the carbon oxysulphide reacts with water, giving carbon dioxide and hydrogen sulphide, so that the final action is—



When perthiocyanic acid is heated with 60 per cent sulphuric acid, a similar hydrolysis must also take place; a certain amount of the thiourea, however, escapes transformation, but the sulphur and carbon oxysulphide are oxidised by the sulphuric acid, sulphur dioxide and carbon dioxide being produced.

The thiourea found among the products of the action of strong sulphuric acid on potassium thiocyanate, is, without doubt, formed in this way by the action of the strong acid on the perthiocyanic acid first produced.

DISCUSSION.

In reply to questions from Mr. Groves and the President, Mr. STEVENS stated that they had not been able to analyse the liquid supposed to be hydrogen disulphide, but they were satisfied as to its identity from a comparison of its properties with those of hydrogen disulphide specially prepared for the purpose.

*51. "*The Composition of Cooked Fish.*" By KATHARINE I. WILLIAMS.

Twenty-two species of fresh fish and five species of preserved fish and oysters were examined after cooking.

Determinations were made of the following constituents:—Water; carbon and hydrogen; nitrogen (total) by Ruffie's method; nitrogen by soda-lime combustion; ash; sulphur; phosphorus; fat; proteids; carbohydrates convertible into glucose; nitrates extracted by dilute alcohol; heats of combustion. The results are recorded in a series of tables.

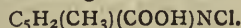
DISCUSSION.

In reply to questions from Mr. Groves, Mr. Hehner, Mr. Sutherland, Mr. Cassal, and Prof. Dunstan, Miss WILLIAMS stated that the fish, in each case, had been analysed in the condition in which it would be eaten. Details of the mode in which the fish had been prepared were given in the paper. The common opinion that fish

contained much phosphorus seemed to be erroneous. Little or none of the phosphorus would be removed in cooking.

*52. "On the Oxidation Products of α -Dimethyl- α' -Chloropyridine." By EMILY ASTON, B.Sc., and J. NORMAN COLLIE, Ph.D., F.R.S.

The present communication is an account of the continuation of some work of one of the authors with A. P. Sedgwick (*Trans.*, 1895, lxvii., 399). The substance α -dimethyl- α' -chloropyridine was obtained by the action of phosphorus pentachloride on pseudolutidostyryl. When oxidised with potassium permanganate, two isomeric acids are obtained, each having the formula—

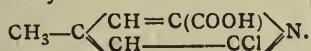


One (m. p. 98°) is much more soluble in water than the other and crystallises with 1 mol. of water; it produces with ferrous sulphate an orange-brown colouration. When strongly heated, it decomposes with much charring and evolution of carbon dioxide and some hydrogen chloride; the residue furnished α -chloro- γ -methylpyridine,—

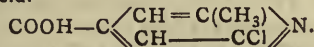


Prolonged treatment with tin and hydrochloric acid gave γ -methylpicolinic acid, and this substance when distilled yielded picoline or γ -methylpyridine.

This acid (m. p. 98°) is, therefore, α -chloro- γ -methyl- α' -pyridine carboxylic acid.



The second acid obtained by the action of potassium permanganate on α -dimethyl- α' -chloropyridine is much less soluble in water. It melts at 214° , and on heating completely decomposes without forming chloropicoline. With ferrous sulphate it furnished a precipitate instead of a colouration. Tin and hydrochloric acid only reduced it very slowly, and it was found to be impossible to separate the chlorine free acid from the unchanged compound. A small quantity of free α -picoline was, however, obtained by distilling the impure reduced acid, thus proving that this acid (m. p. 214°) was the α -chloro- α -methyl- α' -pyridine carboxylic acid.



Attempts were made to obtain the dicarboxylic acid from both the acids by oxidising with potassium permanganate, but without success.

NOTICES OF BOOKS.

The Carbohydrates of Wheat, Maize, Flour, and Bread; and the Action of Enzymic Ferments upon Starches of Different Origin. By W. E. STONE, Ph.D., Professor of Chemistry, Purdue University. Washington: Government Printing Office. 1896.

THE investigations described in this report constitute part of the enquiries being carried on to enable the Secretary of Agriculture to investigate and report on the nutritive value of various articles of food.

It has long been customary to estimate the quantity of carbohydrates in grains and flours by difference, that portion of the material not found to be of the nature of fat, ash, moisture, fibre, or of nitrogenous character, being regarded as carbohydrate nature. The investigations here recorded have been made with the object of discriminating between the various carbohydrates known to be present in cereals, and to trace the effect of the separation of the grain into its parts, as occurs in milling. The different kinds and qualities of wheat, maize, and flour examined, as well as the methods employed, are then fully gone into, and it is important to note that each con-

stituent has been actually determined, and that no result has been obtained by "difference," each carbohydrate, with the exception of cellulose, having been brought to the form of reducing sugar, in which state it was titrated with Fehling's solution.

Both classes of wheat examined were found to contain small amounts of sucrose, ranging from 0.5 per cent upwards; the principal carbohydrate present is starch, which reached 30 per cent. This proportion was considerably increased by milling, which, by eliminating a good deal of the fibre, brought the amount of starch up to 35, and even 45 per cent.

Breads made from these flours were then examined, and, contrary to expectation, it was found that the action of bread-making and baking does not change the nature or condition of the carbohydrates of wheat and maize to any great extent; in the case of wheat flour with one apparently abnormal exception, not more than 10 per cent of the total starch originally present was changed in any way.

The attention of investigators has of late been directed to the action of enzymes on carbohydrates. Recent researches show that different yeasts produce certain specific enzymes, each with an ability to convert some particular class of carbohydrate, such as lactose, maltose, or sucrose; for instance, ordinary yeast inverts the two latter, but not the former, while others will produce enzymes converting either sucrose or lactose, but not maltose. The susceptibility of starches to the more important enzymes, viz., those occurring in grains, or more especially diastase, those occurring in saliva, particularly ptyalin, and those occurring in the pancreatic secretion, were the subject of the next studies. In addition, a few experiments were made with a new diastatic enzyme developed by the fungus *Eurotium oryzae*, discovered by Mr. Jokichi Takamine, and known commercially as "Taka-koji" or "Taka-diastase." The results have a practical bearing on the comparative digestibility of different starches. The action of diastase on starch is increased by minimal amounts of acids, and, on the other hand, it is checked or altogether stopped by greater amounts of acids, and the smallest amounts of alkalis, or alkaline salts.

The ability of diastase to convert large amounts of starch into soluble compounds is remarkable; some authors estimate it at as much as 200,000 times its own weight. There has been much discussion as to the nature and number of the intermediate products of the decomposition of starch; but it is now considered probable that they consist of the various forms of dextrin, which gradually become changed to maltose and isomaltose. Under precisely similar conditions the action of different enzymes on different starches varies considerably, some starches requiring eighty times as long as others for complete saccharification, but they all preserve the same relative order with regard to the commoner enzymic ferments; with taka-diastase, potato-starch was completely converted in seven minutes, this being much quicker than with any of the others; and it is reasonable to assume that the relative degree of susceptibility exhibited by starches in the experiments described would still hold good when subjected to the same enzymes in the process of digestion; in fact, we understand that "taka-diastase" is being largely and increasingly used in cases of dyspepsia with excellent results.

Commercial Fertilisers and Chemicals, Inspected, Analysed, and Admitted for Sale, in the State of Georgia, up to September 1st, 1896. By Dr. GEORGE F. PAYNE, State Chemist. Atlanta, Georgia: G. W. Harrison, State Printer.

THIS book is chiefly devoted to a recital and exposition of the State laws of Georgia regulating the sale of manures. These laws certainly do not err on the side of undue lenity. All manures have to be sold under an

analysis, setting forth the moisture at 212° F., the insoluble phosphoric acid, the available phosphoric acid, the ammonia actual and potential, and, lastly, the potash.

The fact that the purchaser waives the inspection and official analysis "shall be no protection to any person selling or offering fertilisers for sale." In addition to the labels attached to the packages, there are to be fixed on each box, barrel, &c., leaden tags numbered progressively. Any tags left in possession of the manufacturer or merchant at the close of the season shall not be used for another season, nor shall they be redeemable by the Department of Agriculture. The analysis of the State chemist is to be held by an official known as the "ordinary."

The analysis given by the State chemist or his substitute is held to be conclusive evidence against a charge of "partial or total failure of consideration." Hence it seems that if the dealer's analysis is fully confirmed by the State chemist, the dealer is not, as a matter of course, entitled to his money. This, however, is still more equitable than our English practice. With us a purchaser of manures may ignore for a year or more all applications for payment, and at last, when he finds that his creditor is in earnest, may still be allowed to plead defective quality—a plea which, if well founded, should have been urged in answer to the first application for payment.

Agricultural Journal, published by the Department of Agriculture, Cape of Good Hope. January 14, 1897. Cape Town: Townhend, Taylor, and Snashall.

THIS issue is very rich in useful observations. Across the Free State border there are several farms being visited by rinderpest, whilst the colonial area is still entirely free. Inoculation with garlic in the dewlap has proved useless, as has also drenching with petroleum and carbolic acid.

In the Richmond district droves of wild ostriches have made their appearance, and are doing damage.

The vapour of carbon bisulphide is strongly recommended for dealing with destructive insects, care being of course taken to keep lighted candles and matches at a distance.

In South Africa, as far as it is known, there are no soils rich in potash.

Dr. R. Morloth strongly recommends basic slags for supplying the needful phosphate on the Cape Flats and the whole of the Western province.

The Law and Practice of Letters Patent for Inventions.

By LEWIS EDMUNDS, D.Sc., Q.C., of the Inner Temple and of the Oxford Circuit. Second Edition, by T. M. STEVENS, D.C.L., of Gray's Inn and of the South-Eastern Circuit, Barrister-at-Law. London: Stevens and Sons, Limited. 1897. 8vo., pp. 943.

A WORK of this kind presents to the authors unusual difficulties, on account of the complicated nature of the subject matter which lies on the borderland between law and technology, whether the invention relates to physics, chemistry, or mechanics.

A further complication is that such works are rightly expected to deal with Patent Law and Practice as at present existing, whilst the inventor not improbably and not unreasonably demands to see where such law and practice might be improved to the encouragement of our national industries. The Patent Law Amendment Acts of 1852 and 1883 have involved important alterations, both in law and practice, which the authors have set forth fully and clearly.

One of the great defects of our patent system is that it is not imperial. To obtain the protection of an invention for the whole of Her Majesty's dominions costs, if we remember rightly, about £1100. Now, what we want is a system of imperial patents, obtainable on equal terms,

and with equal advantages, in London, Calcutta, Cape Town, Sydney, or other suitable central cities.

Another defect in our patent arrangements is the very "one-sided reciprocity" with alien countries which we have agreed to. Thus a German or a French subject can obtain and uphold a British patent just as easily as a British subject. But if the latter applies in Berlin for the protection of an invention, his application, after prolonged correspondence, may be refused.

In most countries a patent is granted to any applicant *inter alia* on the condition that it is actually and continuously worked on a commercial scale in the country or countries in which it is patented. In Britain no such stipulation prevails, and an article made under any existing and unexpired British patent may be imported from abroad.

A deficiency in the British patent system is that it protects the so-called invention and sale of quack medicines, foods, cosmetics, &c., in which the inventor claims to have some exclusive right or secret. Here we might advantageously copy the German patent law, which refuses patents for medicines, &c. But we might justly and wisely go a step further, and cut off the stratagem by which a German quack evades the law, *i.e.*, by refusing protection to methods for manufacturing "nostrums" or secret and "proprietary" articles whatever.

The work before us will be found invaluable to patentees, counsel, solicitors, patent agents, and all persons who have to take into consideration the value of an invention. We do not hope that it may be of service to the pseudo-inventors—"sifters" as they are called in the manufacturing districts—who are always trying how near they can sail to some valuable novelty.

CORRESPONDENCE.

A RECLAMATION.

To the Editor of the Chemical News.

SIR,—My attention has been called to "A Reclamation" in the *CHEMICAL NEWS* of March 19th (vol. lxxv., p. 134). This note conveys an erroneous impression. The author of it, who is apparently Kippenberger (not Riffenbach) is made to say that "he published this research by Gomberg in the *Zeitschrift für Analytische Chemie*, 1896, p. 466, as a supplement."

On reference to this article I find that Kippenberger, in a "Nachtrag" to the article cited refers to the article of Gomberg and comments on it at some length, but nowhere charges that Gomberg did anything improper. Anyone reading the note in the *CHEMICAL NEWS* would, I think, conclude that Gomberg stole the article, and that the *Journal of the American Chemical Society* had no right to publish it. This, I think, does injustice both to Gomberg and to the Society, and I wish to correct this false impression.—I am, &c.,

EDWARD HART,
Editor of the *Journal of the
American Chemical Society.*

Easton, Pa., April 6, 1897.

Physiological and Pathological Action of the X Rays.—M. Sorel.—The X ray exerts a powerful action upon the cell and its contents, and its prolonged application would be imprudent, at least in certain subjects, near the important organs such as the stomach, the heart, the lungs, and the eyes. In a great number of cases the body of a dead animal is always much more opaque to the X rays than the body of a similar animal immediately after death and still warm.—*Compt. Rend.*, cxxiv., No. 15.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 15, April 12, 1897.

Law of the Discharge of Electrified Uranium into the Air.—Henri Becquerel.—The author gives in this paper the law of the loss of electricity by uranium as a function of time and of the potential of the electrified bodies. Salts of uranium which he has preserved for more than a year protected from any known radiation continue to emit, with an intensity scarcely decreased, radiations which produce photographic impressions through opaque bodies.

Photography of König's Flames.—M. Maraze.—This paper requires, or rather consists of, eight photographic proofs.

Experiments made with a New Kathodic Apparatus generating X Rays, and with several Vessels sealed on the same Gaseous Circuit.—Foveau de Courmelles and G. Segny.—On observing the luminous, fluorescent, and cathodic effects produced we find that the internal pressure in an exhausted tube is not equal at all points; that towards the extremity the vacuum is much more complete than at the other extremity of the same gaseous circuit, and that the molecules which are able to remain in such highly rarefied media are driven with extreme violence towards the most extreme point of the circuit of the apparatus. The authors signalise the rapidity and definiteness of the results obtained with the vacuum tube.

Formation Heats of Formic Aldehyd.—Marcel Delépine.—A thermochemical memoir. The formation heat of gaseous formic aldehyd from its elements is +25.4 cal., and of dissolved aldehyd is +40.4 cal.

Formation of Ammonium Cyanide and its Manufacture.—Denis Lance.—Ammonia gas passing over carbon at a temperature between 1000° and 1100° C. always yields ammonium cyanide. The yield of cyanogen is more considerable if we use a mixture of ammoniacal gas, nitrogen, and hydrogen. The yield reaches its maximum at 1100° C., and when the gases are in the following proportions:— $\text{NH}_3 = 1.26\text{th}$ of the mixture formed by N and 10H . In these conditions at least 70 per cent of the nitrogen of the ammonium cyanide is derived from the free nitrogen of the mixture, *i.e.*, from the nitrogen of the air.

MISCELLANEOUS.

The Stas Memorial.—Invitations have been sent by the Organising Committee to all subscribers to attend the Inauguration of the Monument erected to Stas. The ceremony will take place on May 11th, at 4 p.m., in the garden of the Palace of the Academies, Brussels.

Iron and Steel Institute.—The Annual Meeting of the Institute will be held at the Institution of Civil Engineers, Great George Street, Westminster, on Tuesday and Wednesday, the 11th and 12th days of May, 1897, commencing each day at 10.30 o'clock a.m. The following is a list of Papers that are expected to be read and discussed:—

"On the Permeability of Steel-making Crucibles," by Professor J. O. Arnold and F. K. Knowles.

"On the Practice of the Combined Open-hearth Process of Bertrand and Thiel," by E. Bertrand.

"On the Agricultural Value of Sulphate of Ammonia from Blast-furnaces," by F. J. R. Carulla.

"On the Specific Heat of Iron," by Professor W. N. Hartley, F.R.S.

"On Charging Open-hearth Furnaces by Machinery," by Jeremiah Head.

"On the 'Weardale' Re-heating Furnace," by H. W. Hollis.

"On the Effect of Phosphorus on Cold Shortness," by Baron Hanns Juptner von Jonstorff.

"On the Determination of Hardening and Carbide Carbon," by Baron Hanns Juptner von Jonstorff.

"On Malleable Cast Iron," by G. P. Royston.

"On Carbon Changes connected with Malleable Cast Iron," by G. P. Royston.

"On Microscope Accessories for Metallographers," by J. E. Stead, Member of Council.

"On Central Blast Cupolas," by T. D. West.

Silver Hydride.—Edwin J. Bartlett and W. F. Rice (*Am. Chem. J.*, xix., 49-52).—Silver hydride, AgH , was prepared by precipitating a dilute solution of silver nitrate with dilute hypophosphorous acid in excess. The solution becomes wine-coloured at first, changing to black, and after a few minutes black spongy flakes are precipitated, which are filtered at once. The filtrate, on long standing or boiling, deposits metallic silver. Silver hydride is not decomposed by water.—*Journ. American Chemical Society.*

Paraisobutylphenoxyacetic Acid.—W. P. Bradley and F. Kniffen (*Am. Chem. Journ.*, xix., 70-76).—Paraisobutylphenoxyacetic acid was prepared by heating paraisobutylphenol and chloroacetic acid dissolved in sodium hydroxide. The mixture was neutralised with sulphuric acid, treated with an excess of sodium carbonate, and extracted with ether to remove any excess of phenol. The resulting liquid was then evaporated to a small bulk, acidified, and the acid extracted with ether. The compound is a cream-white solid, melts at 86.5°, and crystallises from ligroin in radial crystals. The barium and magnesium salts crystallise well. The amide, prepared from the methyl ester, crystallises from ligroin in white plates, which melt at 134°. The anilide, metanitrilide, ortho- and paratoluides, and hydrazide are described. A tetranitro derivative of the anilide was formed by the action of fuming nitric acid. The fact that the four nitro-groups are divided equally between the two benzene rings was proved by the action of potassium hydroxide, which decomposed the compound into the ortho- and paradinitraniline.—*Journ. Amer. Chem. Soc.*

MEETINGS FOR THE WEEK.

MONDAY, 3rd.—Society of Arts, 4.30. (Cantor Lectures). "Design in Lettering," by Lewis Foreman Day.

Royal Institution, 5. General Monthly Meeting.

TUESDAY, 4th.—Royal Institution, 3. "Volcanoes," by Dr. Tempest Anderson, B.Sc.

Society of Arts, 4.30. "The Arctic and Antarctic," by Aubyn Trevor-Battye.

WEDNESDAY, 5th.—Society of Arts, 8. "The Railway to India," by C. E. D. Black.

THURSDAY, 6th.—Royal Institution, 3. "Liquid Air as an Agent of Research," by Prof. Dewar, F.R.S., &c.

Chemical, 8. Ballot for the Election of Fellows.

"A Bunsen Burner for Acetylene," by A. E. Munby, M.A.

"Reactions between Lead and the Oxides of Sulphur," by H. C. Jenkins and E. A. Smith.

Society of Arts, 4.30. "Kafiristan—its Manners and Customs," by Sir George Scott Robertson, K.C.S.I.

FRIDAY, 7th.—Royal Institution, 9. "Romance," by "Anthony Hope."

SATURDAY, 8th.—Royal Institution, 3. "The Greek Theatre according to Recent Discoveries," by the Rev. J. P. Mahaffy, D.D.

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1954.

RECENT DETERMINATIONS OF THE ELECTRICAL CONDUCTIVITY OF ALUMINIUM.

By JOSEPH W. RICHARDS and JOHN A. THOMSON.

MANY and various values have been determined for the electrical conductivity of this metal. The causes have been as follows:—

(1). The impurity of the metal used. Until 1886, the best commercial aluminium rarely surpassed 98 per cent in purity, and it was not until 1889 that commercial metal of 99 per cent was put on the market. As will be shown later, the effect, even within these narrow limits, is to change the conductivity nearly 10 per cent.

(2). The reference of the conductivity to copper or silver as standards. In such cases, the exact purity of the copper or silver and the physical condition of these metals, whether hard or soft, must be known in order to give the comparison its proper value; but these were in most cases either unknown or neglected. Even at the present time, the absolute conductivity of pure soft copper or silver cannot be said to be fixed closer than within 1 per cent, so that figures for conductivity of aluminium, given only with reference to copper or silver, cannot, at best, have an accurate significance.

(3). Lack of an accurate standard of absolute resistance. The adoption of standard units of resistance, by international concert, and the consequent multiplication of registered copies, has made it an easy matter to use in experiments certified instruments of accurately known resistance, and thus to dispense with self-constructed units of comparison in favour of more accurate standards.

(4). Imperfect methods of measurement. Of late years, several ingenious arrangements have been devised for eliminating from the calculations of experiments the resistance of connections, always an uncertain quantity, and more refined instruments for measuring and balancing electric currents have been constructed, thus permitting of increased accuracy in results.

In the following experiments the specimens tested were kindly furnished by the Pittsburgh Reduction Company, and were all analysed by Mr. Handy, of the Pittsburgh Testing Laboratory, so that their composition was accurately known. The conductivity is given in absolute measurement, so that no reference to any other metal as a standard can affect the results. This was rendered possible by the use of a certified standard resistance coil of 1 "International" ohm, whose possible error is not over 0.02 per cent, and by the use of the *Carey Foster* method of comparison. The metal was in wire, of 50-foot lengths, the diameter of which was measured by a micrometer and checked by weighing and determining the specific gravity. The wires were wound on wooden bobbins and immersed in oil, the temperature of which

was given by a thermometer. The galvanometer used was a reflecting instrument, sufficiently delicate for all purposes. The standard coil was immersed in water, and the room was kept at a constant temperature. The bridge wire used was carefully calibrated, and all readings were taken several times. Two separate wires were tested in case of Experiment 1, the result given being the mean of two results, which differed only one-hundredth of 1 per cent from each other.

For the reduction from the working temperature to 0° C. an experiment was made with wire No. 1, which showed that between 27° C. and 0° C. its temperature coefficient was 0.00392 per degree. This coefficient was used for the nearly pure wires, while for 4 and 5 a slightly lower coefficient, determined by Mr. Scott, was used. It appears that the purer the metal the greater its temperature coefficient.

Conductivity tests of a similar set of wires were made by Mr. C. F. Scott, electrician of the Westinghouse Electric Company, Pittsburgh. They were made by comparison with pure copper, with a Wheatstone bridge. These results can only be compared with ours by assuming a certain value for the conductivity of copper, and even then we cannot say how nearly the copper used by Mr. Scott would approach that standard. Sir W. Thompson's value for the specific resistance of copper is 1580, Dewar's 1562. In the following table we reduce our results to each of these standards, and add Scott's results for comparison:—

Relative Conductivity (Copper = 100).

	Richards and Thomson. Using for copper the resistances		C. F. Scott. Actual resistance of copper employed not known.
	(1580)	(1562).	
1. Soft	65.0	64.2	—
1. Hard	64.4	63.7	63.1
2. Soft	62.3	61.6	—
2. Hard	61.1	60.5	62.2
3. Hard	55.5	54.9	56.2
4. Hard	56.0	55.4	58.5
5. Soft	52.9	52.3	—
5. Hard	52.5	51.9	55.0

Temperature Coefficient for 1° C.

	C. F. Scott. (Between 15° and 80° C.).	Richards and Thomson. (Between 0° and 27° C.).
1.	0.00385	0.00392
2.	0.00385	—
3.	0.00360	—
4.	0.00361	—
5.	0.00359	—

In connection with the results of Mr. Scott and ourselves, we may mention for comparison those of Charpentier-Page, who used what he calls *pure* aluminium, which may safely be assumed to be the No. 1 grade of European aluminium, averaging 99 per cent pure. He finds as follows:—

ANALYSIS.					Resistance at 0° C. of a wire 1 metre long by 1 m.m. diameter, in ohms.	Specific resistance at 0° C., i.e., resistance of 1 c.c. in absolute (C.G.S.) units of resistance.		
Aluminium.	Iron.	Copper.	Silicon.	Sodium.		Hard.	Hard.	Annealed.
1. 99.66	0.10	0.00	0.16	0.008	—	0.031245	2453.7	2432.2
2. 99.58	0.25	0.00	0.16	0.052	—	0.03290	2584.0	2535.0
3. 98.77	0.20	0.57	0.45	0.012	—	0.03627	2848.0	—
4. 97.16	0.25	2.26	0.30	0.032	—	0.03590	2819.6	—
5. 94.39	0.25	7.07	0.24	0.052	1.50	0.03583	3011.4	2984.7

	Specific resistance (Calculated to 0° C.).	Compared with copper.	
		(1580). Per cent.	(1562). Per cent.
Soft ..	2659	59.4	58.8
Hard..	2684	58.9	58.2

It should be noticed that these results fall exactly between our Nos. 2 and 3, also just where its composition would most probably lie. The results also agree closely with ours in showing almost exactly 1 per cent greater conductivity for the annealed than for the hard-drawn wire.

Dewar and Fleming have also recently found as the specific resistance of "Swiss aluminium about 99 per cent pure" the value 2563 at 0° C., which is 60.9 per cent of that of copper, according to their own measurements. This also fits in well with our determinations, but the comparison would have been much more satisfactory if the exact composition of their metal had been determined.

C. K. McGee determined, in 1890, the conductivity of aluminium analysing 98.52 per cent pure to be 54.8 per cent that of copper when unannealed. This metal was nearly identical with our No. 3 in composition, and the results are the same within 1 per cent.

The conclusions we would draw from these experiments and comparisons are that—

The conductivity of hard drawn commercial aluminium is strongly affected by impurities, being, approximately,—

	(Copper=100).
98.5 per cent pure aluminium ..	55.0
99.0 " " ..	59.0
99.5 " " ..	61.0
99.75 " " ..	63.0—64.0
100.0 " " ..	probably 66.0—67.0

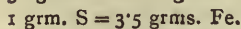
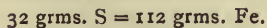
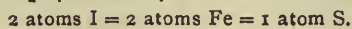
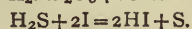
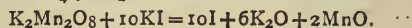
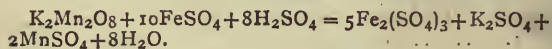
Annealed wire has a conductivity very nearly 1 per cent greater than the unannealed.—*Journal of the Franklin Institute*, March, 1897.

STANDARD IODINE SOLUTION FOR SULPHUR DETERMINATIONS.

By EDWARD K. LANDIS.

THE following calculation shows an easy method of preparing Payne's iodine solution, with the least amount of calculation.

Reactions.



When 5 grms. are taken for analysis, 0.01 per cent = 0.005 grm., and this multiplied by 1000 = 0.5 grm. in a litre.

Let x = value of 1 c.c. $K_2Mn_2O_8$ in Fe in grms.

$$\text{Then } \frac{0.5}{x} = 0.5 \times \frac{3.5}{x} = \frac{1.75}{x}.$$

Therefore 1.75 divided by the value of one c.c. potassium permanganate in iron in grms. gives the number of c.c. of potassium permanganate to be added to the potassium iodide and sulphuric acid and diluted to one litre, to form iodine solution of such strength that one c.c. will be equal to 0.01 per cent sulphur when using five grms. of sample.—*Journ. American Chem. Soc.*, xix., No. 3.

SOME EXPERIMENTS WITH CATHODE RAYS.*

By A. C. C. SWINTON.

THE extensive employment of the focus form of Crookes tubes as the most efficient known means of generating X-rays, has rendered advisable the more complete investigation of the cathode ray discharge in tubes of this description.

Hitherto, the usual method of investigating the characteristics of a cathode ray discharge apart from its mechanical properties, and beyond what is visible to the unassisted eye, has been by allowing the rays to fall upon a screen of some brightly fluorescent material, such as glasses of various descriptions, or screens covered with fluorescent salts. With all of these the maximum amount of fluorescence appears to be produced by such comparatively weak cathode rays, that in some cases the special effects produced by the more powerful rays seem to be more or less entirely masked, while the well-known phenomenon of the fatigue of fluorescent substances, when exposed to the more active rays, conduces to the same result.

Surface Luminescence of Carbon when exposed to Cathode Rays.

I have found in some cases that by replacing the usual screen, made of or covered with fluorescent material, by one of ordinary electric light carbon, much appears which was previously invisible. When a concentrated stream of powerful cathode rays are focussed upon a surface of carbon in this manner, a very brilliant and distinctly defined luminescent spot appears on the surface of the carbon at the point of impact of the rays, the remainder of the carbon remaining black. This luminescent spot seems to have a very close relation to the fluorescent spots on glass and on other fluorescent materials under similar influence. The effect is evidently a purely surface effect, as when the cathode stream is rapidly deflected by means of a magnet the luminescent spot on the carbon moves with no perceptible lag. Further, though, as is also the case with glass, the whole of the carbon becomes gradually heated to a considerable extent if much power be employed for a long period of time, these luminescent spots are instantaneously produced on carbon of very considerable brilliancy with but a comparatively low power. Again, just as glass is known to become fatigued under the influence of cathode rays, so that after a time it refuses to fluoresce so brightly as before, so carbon is similarly fatigued, though only after having been very strongly acted upon. Carbon, like glass, also recovers its property of giving a surface luminescence to some extent, though it does not seem to entirely recover, at any rate, at all rapidly.

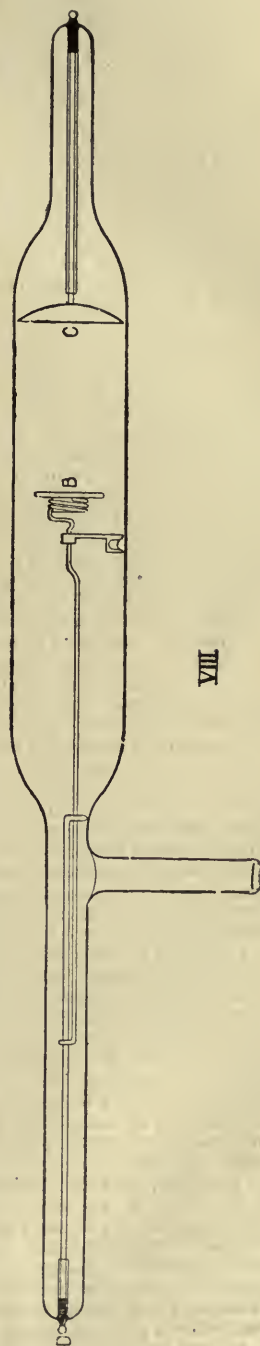
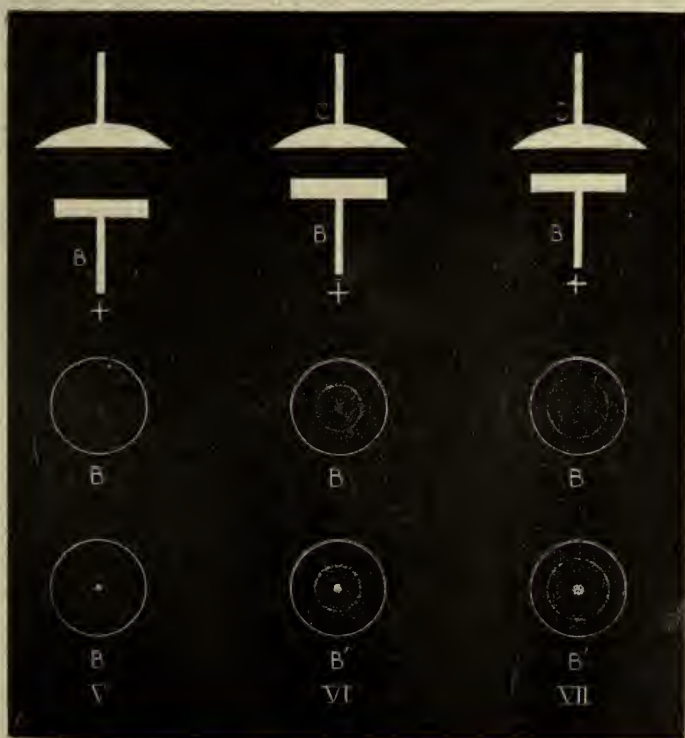
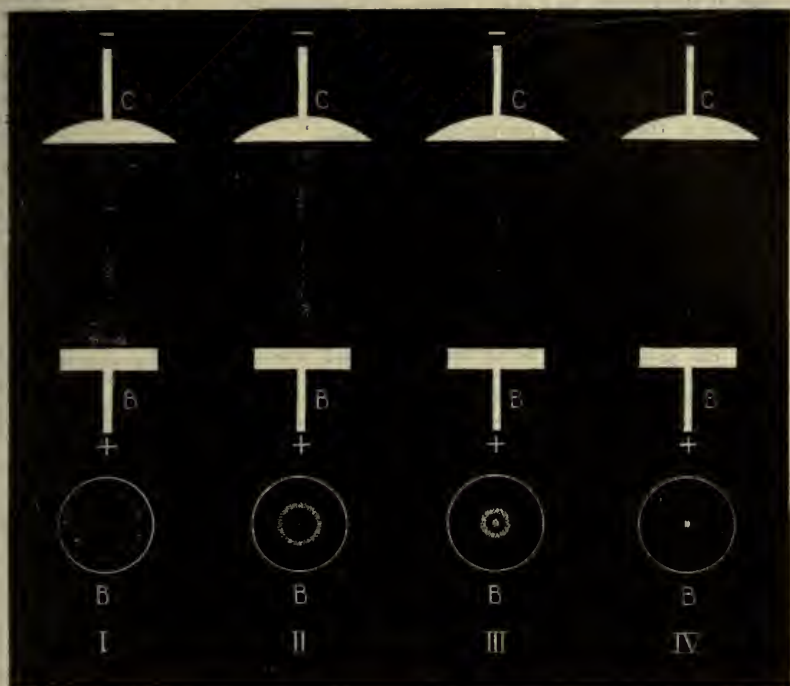
That the rays which produce the luminescence of the carbon are the same rays that cause fluorescence of the glass can be proved by deflecting the rays from the carbon on to the glass by means of a magnet.

As it is exceedingly difficult, if not impossible, to obtain carbon plates which do not contain hydrocarbons and other volatile matter which are rapidly given off and reduce the vacuum very quickly when the carbon becomes at all heated, it is necessary to keep the tube connected to the mercury pump, so that the vacuum can be restored after each experiment. This arrangement was followed in all the experiments described below, except where specific mention is made to the contrary.

Apparent Form of the Cathode Ray Discharge in a Focus Tube.

As is well known, in tubes of the ordinary focus type with a single spherical concave cathode, the rays coming

* A Paper read before the Royal Society, March 11, 1897.



off normally to the cathode surface appear to converge in more or less of a cone to a focus, and, if the vacuum be not too high, to diverge again immediately in another cone upon the other side of the focus. At higher vacua the rays, after passing the focus, do not appear to diverge again at once, but seem to form themselves into a description of thread which connects the convergent and divergent cones, and is longer or shorter according as the vacuum is higher or lower. The angle of the divergent cone appears, however, to be always proportional to that of the convergent cone. The focus, or perhaps more correctly the point at which this thread commences, seems always to be more distant from the cathode than the centre of curvature of the latter, but the variation in this respect seems to be less and less the higher the exhaustion. This is no doubt due to the mutual repulsion of the rays, and accords with the assumption that the rays consist of charged particles, which travel more and more rapidly the higher the exhaustion. Probably for the same reason cathodes that are only slightly concave focus further in proportion beyond their centres of curvature than do deeply concave cathodes for the same vacuum.

Apparent Hollowness of the Divergent Cone of Rays.

When the divergent cone is thrown upon a thin platinum disc, as in the ordinary focus tube, and sufficient electric power—say, from a 10-inch Ruhmkorff coil—is employed, the platinum quickly attains to a red heat. With platinum, either the whole disc becomes uniformly heated, or in the event of the diameter of the cone of rays where it strikes the platinum being small, compared with the area of the platinum, that portion of the platinum covered by the base of the cone becomes uniformly heated to a higher temperature than the remainder. This is as much as can usually be seen with platinum, though rather more is sometimes visible with aluminium; but if, instead of either metal, the disc is made of ordinary electric light carbon, I have found that the luminescent portion of the carbon, instead of comprising the whole disc, or consisting of a uniformly heated circle, will in some cases take the shape of a brilliantly luminescent and apparently white hot ring, with a well-defined dark, and seemingly quite cold, interior. As the dimensions of the cone of rays are increased or decreased by decreasing or increasing the vacuum, the luminescent ring will be found to increase or decrease correspondingly in diameter, at the same time being brighter when small than when large. Further, when the ring is very small it will usually have a very brightly luminescent central spot, with a dark intervening portion between this spot and the ring, and when the vacuum is further increased the ring will gradually close in upon the spot until only the latter remains.

Figs. 1, 2, 3, and 4 show diagrammatically these hollow effects, as produced by spherical aluminium cathodes, 1·125 in. diameter and 0·708 in. radius of curvature, for four degrees of vacuum, 1 being the lowest and 4 the highest exhaustion. The upper portion of each of these figures represents the general appearance of the cathode discharge between the spherical concave aluminium cathode C at the top, and the carbon anti-cathode B at the bottom, as accurately as it is possible to represent evanescent coloured appearances in monochrome. The other appearances, due to the dark space and fluorescence of the glass, are omitted for the sake of simplicity. Beneath each of the elevational views of the cathode discharge will be found a plan view of the carbon anti-cathode, showing for each condition of vacuum the effect of the cathode discharge upon the carbon anti-cathode, in forming a brightly luminescent hollow ring, gradually decreasing in diameter as the vacuum is increased, until it centres on a point, as already mentioned.

It may further be remarked that the diameter of the luminescent ring may be increased or diminished, or finally reduced to a point, without altering the degree of vacuum, by moving the anti-cathode away from or towards or finally into the focus of the cathode stream, the

appearance of the ring in each of these cases being practically similar to those shown in the figures for a uniform distance with varying vacuum. When the anti-cathode surface is not at right angles to the line of the discharge, the ring, in place of being circular, takes the proper form of a conic section. The holding of a magnet near the tube distorts the ring from a circular shape and moves its position on the carbon.

From these experiments it appears that the divergent cone of cathode rays acts as though it were not of uniform density throughout its section, but, at any rate, in some instances as if it were completely hollow. This fact does not appear to have previously been noted.

Apparent Hollowness of the Convergent Cone of Rays.

The apparent hollowness of the divergent cathode ray being thus established, it was thought desirable to ascertain whether the same condition of affairs exists in the converging beam of rays between the cathode and the focus. Owing to the well-known difficulty of getting any discharge to pass when the distance between the electrodes is less than the thickness of the dark space, and to the disturbing effect which the anti-cathode screen is found to have when brought within the focus of the cathode, especially with high vacua, this question was found much more difficult to decide than that of the hollowness of the divergent cone. However, that the convergent cone also acts under certain circumstances as though it were almost completely hollow, and acts generally as if it had a considerable tendency towards hollowness at low vacua, was also finally fully determined. The lower portions of figs. 5, 6, and 7 show the bright ring appearance upon the carbon anti-cathode at two different degrees of exhaustion, B being higher vacua than B', and with the anti-cathode at the three different distances from the cathode within the focus of the latter, as shown in the upper part of each figure. As will be observed in this case, the degree of vacuum is found not to appreciably affect the dimensions of the figure, though it should be stated that the vacua in each case were comparatively low, as vacua as high as those employed when the anti-cathode was outside the focus gave no results at all. As will be seen, however, the diameter of the luminescent ring is affected by the degree of proximity of the anti-cathode to the cathode, being larger when the distance is small than when it is great; while in every case there is a decided tendency towards hollowness, though usually with some slight internal luminescence and with a bright central spot, while in one case, when the anti-cathode was very close to the cathode and the vacuum was comparatively high, the ring is seen completely hollow, and there is no central spot.

A convenient form of tube for showing the apparent hollowness of both the divergent and convergent cone of cathode rays is shown in fig. 8, where the anti-cathode disc B, made of electric light carbon, is supported upon a small carrier which slides upon the bottom of the tube, and is connected to the anode terminal, D, by means of two aluminium wires, each of which have a ring at their extremity through which they respectively pass. As the carbon, under the action of the cathode rays, gives off hydrocarbon vapour, it is necessary, as already mentioned, to try all these experiments with the tube connected to the mercury pump; but with this connection made through a slightly flexible mercury joint it is possible, by inclining and gently tapping the tube, to bring the anti-cathode to any desired position either near or far away from the cathode. For experiments upon the divergent cone, it is not necessary that the anti-cathode screen should be connected to the anode terminal, and, consequently, the sliding aluminium wires inside the tube are not required. They are, however, necessary when observations are to be made on the convergent cone between the cathode and the focus, as the anti-cathode screen when placed within the focus must be connected to the anode, or it appears to

get negatively charged, and acts itself as an additional cathode, throwing cathode rays in all directions.

It may here be mentioned that the fatigue of the carbon already alluded to renders necessary some precautions in carrying out the above-mentioned experiments, as otherwise the observer may be misled into thinking that a beam of cathode rays is effectively hollow when this is not the case, owing to the centre of the carbon covered by the beam having been fatigued by some previous experiments. By taking the precaution, however, of deflecting the cathode beam by means of a magnet on to various portions of the carbon screen, such errors may be avoided. It should also be noted that these hollow effects appear only to be obtained with fairly short focus cathodes, such as are usually employed in X-ray focus tubes, that is to say, with cathodes whose diameter is large as compared with their radius of curvature, so that the rays converge and diverge rapidly to and from the focus. With comparatively flat, long, focus cathodes, the cones do not show any signs of being hollow, and produce a uniformly luminescent spot upon the carbon of larger or smaller diameter, according to the conditions of vacuum and the position of the screen.

For instance, while cathodes 1·125 inches diameter and 0·708 inch radius of curvature gave in the manner described distinctly hollow convergent and divergent cones, a cathode 1 inch diameter and 1·5 inches radius of curvature gave convergent and divergent cones that appeared to be uniformly solid under all conditions.

(To be continued).

THE QUALITATIVE SEPARATION OF ARSENIC, ANTIMONY, AND TIN.*

By S. G. RAWSON, D.Sc., F.I.C.,
Lecturer in Chemistry, Technical College, Huddersfield.

To any originality in the process which I propose describing I can lay no claim, the principles involved depending upon a combination of certain well-known methods. The suggested separation of these metals is based primarily upon the use of oxalic acid as originally published by Clarke in his paper (CHEMICAL NEWS, xxi., p. 124) upon the quantitative estimation of these metals, a process which to me never seems to have come into the prominence which it so thoroughly deserves. I have but little faith in the methods now employed, especially in those for the separation of antimony and tin. The former metal is frequently evolved either as the hydride or deposited as a black stain upon platinum; the tin being thrown down upon zinc; this deposit is then scraped off, dissolved in acid, and the solution treated with mercuric chloride. That other people share in my disbelief in this method is shown by the fact that, in so far as my knowledge goes, I have never heard in any examination of more than two of those bodies being given for qualitative determination in one and the same substance. Even under these favourable conditions, it is seldom that the mixture contains only a small percentage of one of the constituents; the amounts more usually approximate to equality, the difficulties of separation being thereby much reduced. The method I would suggest is as follows:—The sulphides of the metals are washed upon the filter-paper, and the whole or part of the residue is placed in a test-tube and boiled with 2 or 3 c.c. of concentrated hydrochloric acid, to which a drop or two of nitric acid is added, and again boiled. A yellow residue after the treatment with hydrochloric acid gives a preliminary clue as to the presence of arsenic, this sulphide being slightly, if at all, attacked by the acid. A saturated solution of oxalic acid is added in quantity sufficient to fill two-thirds

of the test-tube; the whole is boiled, and crystals of oxalic acid are added until a hot concentrated solution of oxalic acid is obtained. A stream of sulphuretted hydrogen is passed, the whole of the arsenic and antimony being precipitated as sulphides, which are filtered off, the tin remaining in solution. To the filtrate ammonia is added until distinctly alkaline. If a precipitate should then appear, which will not be the case unless a large amount of tin be present, add ammonium sulphide drop by drop until the precipitate re-dissolves: this it will do very readily. Acidulate with acetic acid; a heavy white precipitate, turning brown, indicates tin as a mixture of oxide and sulphide. It may be here noted that the treatment of ordinary ammonium sulphide with acetic acid produces a precipitate of sulphur, but the appearance of the precipitates formed in the two cases is quite distinct and cannot be mistaken. Turning again to the residual sulphides of arsenic and antimony, these may be treated either with ammonium carbonate in the well-known way, or, and preferably I think, as follows:—Dissolve in hydrochloric acid with two or three drops of nitric acid, boil, and place the solution in a Marsh apparatus. The evolved hydrides are then passed through a solution of silver nitrate, and the antimonide of silver formed filtered off. To the filtrate add a few drops of silver nitrate, and then very cautiously ammonium hydrate until the yellow precipitate of silver arsenite appears. The silver antimonide precipitate is washed, boiled with tartaric acid and filtered, a little hydrochloric acid is added, and sulphuretted hydrogen passed through the filtrate, orange-red antimony sulphide being thrown down. To both of these precipitates the ordinary reduction and sublimation tests can be applied. The above method gives thoroughly good results, and with amounts of the respective sulphides varying within wide limits, and does not require that tinkering with bits of platinum foil and of zinc which is both unreliable and unpleasant.

THE USE OF ALUMINUM FOR CONDENSERS.*

By T. H. NORTON.

IN connection with the extended use of aluminum in this laboratory for various forms of apparatus, water-baths, air-baths, Bunsen burners, hot water filtering funnels, &c., it seemed desirable to study the availability of the metal for condensation processes.

For this purpose a condenser was constructed as follows:—The outer jacket was of glass; the inner tube was of aluminum and possessed the following dimensions—length 122 c.m., external diameter 1 c.m., inner diameter 8½ m.m., weight per metre 29 grms. At a distance of 15 c.m. from the end, the tube was bent at right angles. This permitted of connection with a distilling flask, without allowing the condensing vapours to come in contact with any substance but aluminum. It might be mentioned here that in order to bend an aluminum tube of these dimensions satisfactorily, it is necessary to fill it with molten lead, and further, that several distillations with water are requisite in order to remove completely slight traces of lead adherent to the surface of the aluminum, after this operation.

The method of testing the condenser was to distil a measured quantity of a liquid from the glass flask, used as a still, collect the distillate in glass, evaporate it from weighed platinum dishes, and note the weight of the ignited residue, thus ascertaining whether there was any appreciable attack on the aluminum. Nothing was attempted beyond the ordinary precautions for preventing dust from contaminating the distillates.

* A Paper read before the Society of Chemical Industry (Yorkshire Section), January 25th, 1897.

* Contributions from the Chemical Laboratory of the University of Cincinnati. From the *Journal of the American Chemical Society*, xix., No. 2.

The liquids first employed were organic. In each case 500 c.c. were distilled, and the weights of the residue left on evaporating the distillate noted. The following results were obtained:—

Liquid.	Residue from 500 c.c.
Ethyl alcohol (specific gravity 0.809) ..	0.001 grm.
Benzene	0.0016 "
Nitrobenzene	0.0004 "
Chloroform	0.0002 "
Ethyl ether	0.0000 "
Acetone	0.0001 "

In all these cases it was evident that very rapid distillation could be carried on with an exceedingly short tube, on account of the high conductive power of the aluminum. The residues obtained showed that there was practically no attack upon the aluminum.

The deportment of the metal towards steam was next studied, and here it was deemed wise to establish in all cases comparative experiments with glass and block tin. The glass condenser tube used for this purpose was 84 c.m. long and had an inner diameter of 16 m.m.; the tin condenser tube was 305 c.m. long and had an inner diameter of 21 m.m. With the exception of differences in the superficial surface for condensation, other conditions were essentially identical. Three series of distillations were carried on with the three following samples of water:—

- Hydrant water (Ohio river water), containing much impurity.
- Hydrant water (Ohio river water), containing less impurity.
- Distilled water.

In all cases 500 c.c. were employed. The following residues were obtained:—

	Aluminum.	Block tin.	Glass.
A.. ..	0.0112	0.006	0.0118 grm.
B.. ..	0.0032	0.0028	0.0091 "
C.. ..	0.0035	0.005	0.008 "

A check determination on the amount of dust collecting in the platinum dish during the time for evaporation, showed it to be 0.0002 grm. after ignition.

These results would show that as far as purity of the product is concerned, aluminum possesses about the same advantages over glass as tin, in connection with the distillation of water. In lightness and conductivity it exhibits marked superiority to the tin.

For use with neutral organic liquids it is well adapted, more especially in the distillation of low boiling substances, such as ether. Here, also, the high thermal conductivity, as well as the absence of brittleness, are factors in its favour as compared with glass.

Mr. R. W. Hochstetter rendered valuable assistance in the determination of the above data.

SOIL FERMENTS IMPORTANT IN AGRICULTURE.*

By HARVEY W. WILEY,
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Washington, D.C.

Introductory.—Soil ferments important in agriculture are those which help to make the soil from original rocks and those which are active in preparing the food of plants for absorption and assimilation. The old idea that the soil is an inert mass of mineral matter has given way to the new conception of the soil as a living organism. The parts of the soil which are not endowed with life at the present

time have their highest significance as the environment of the living organisms which they contain, and which they may help to nourish. The plant which forms the growing crop receives its nourishment through the media of the air and soil; but this nourishment must undergo a process of digestion, similar to that suffered by the food which nourishes animals, before it becomes available as plant-food. Indeed, the purely mineral, inorganic foods of plants are probably not always absorbed as such, and must undergo a decomposition before they are assimilated. A striking instance of this is shown in the case of silica, an important plant-food and a type of inert mineral matter. Silica is highly insoluble, and apparently the least suited of the mineral constituents of the earth to enter the vital organism of the plant. Yet not only do we find it in the tissues of the mature plant, but also, strange to say, in the greatest abundance in those parts of the plant organism—viz., the leaves—most remote from the sources of supply. It is evident from this that the highly insoluble silica of the soil must undergo a complete solution in order to be carried by the juices of the plant through the network of cellular tissues, to be finally re-deposited in the leaf.

The part which soil ferments have played in the formation of arable soil from the original rocks is not thoroughly appreciated. The naked rocks of high mountains comprise mineralogical types of the most varied nature, viz., granite, porphyry, gneiss, mica schist, volcanic rocks and limestones of all varieties, and all these have been found to be covered with a nitrifying ferment which is doubtless extremely active in producing incipient decay. At the high altitudes in which these observations have been made, the activity of bacteria is necessarily limited by the low temperature to which they are subjected during the greater part of the year. During the winter season their life is suspended, but is not extinguished, since they have been found living and ready to resume all their activity after an indefinite sleep, perhaps of thousands of years, on the ice of the glaciers, where the temperature never rises above the freezing-point. When the activity of these ferments in the most unfavourable conditions is recognised, it is easily seen how much more active they become when brought down to lower levels, where they are nourished by the favouring conditions which exist, especially during the summer, in cultivated soils. In fact, the importance of the action of these bodies on the mineral particles of which the soil is largely composed has never been fully recognised, and there is no doubt whatever of the great significance of their decomposing action in the liberation of plant-food locked up in undecomposed mineral structures. In this case the activity of the bacteria is not limited to the surface of rock masses, but permeates every particle of soil, and thus becomes effective over a vastly extended surface.

When the extreme minuteness of these organisms, and of the phenomena which they produce, is considered, there may be a tendency to despise their importance; but by reason of the fact that their activity is never ceasing, and of the widest application, it must be placed among the geologic causes to which the crust of the earth owes a part of its actual physiognomy, and to which the formation of the deposits of the comminuted elements constituting arable soil is due.

But the action of these ferments has not stopped with the aid they have given to soil formation. It is highly probable that they assist in a most marked manner in the final dissolution of the soil particles, and the setting free of the plant-foods which they contain. It is quite certain that in the primary decay of bare rocks, especially at high altitudes, the nitrifying organism plays a highly important part, preparing the surface of the rock for the first growth of lichens and other low vegetable organisms, from which the first traces of humus are formed. The discovery that the nitrifying organism can subsist upon a purely mineral food is one of the chief supports of the idea that they were especially active in the very beginning

* Abstract of a Lecture delivered before the Chemical Section of the Franklin Institute, February 16th, 1897.

of soil formation. It has been shown that these bacteria can be developed by absorbing from the ambient atmosphere traces of ammonia and other bodies which may be present in the air. There is thus discovered in the very first products of the attrition of rocks the characteristic element of vegetable soil, viz., humus, the proportion of which increases rapidly with the processes of disintegration, until finally the decaying mass is capable of sustaining chlorophyll-containing plants. Not only upon the surface of exposed rocks have these organisms been discovered, but they are found to extend also to a considerable distance in the interior. They not only play an important part by direct action upon the mineral matters which the rocks contain, but later on, through the production of nitric acid, greatly favour the final solution of the soil particles.

Kinds of Organisms in the Soil.—The nitrifying organisms in the soil exist in common with hundreds of others, many of which are doubtless active upon the soil particles. The organisms to which particular attention is called in this address, in addition to those which help to dissolve the soil particles already mentioned, are those which are active in preparing organic foods for absorption and assimilation by plants, and those which act upon free atmospheric nitrogen and bring it into a shape suited to plant-nutrition.

In general these are called the nitrifying ferments, and their action is uniformly favourable to vegetable growth. Attention should also be given to another class of organisms found in the soil, whose activity is inimical to plant growth or hurtful in some other way. This class comprises the denitrifying organisms, and those of a pathogenic nature which may exist in the soil, and by their activity cause disease in man and beast.

The Nitrifying Ferments.—The micro-organisms of most importance to agriculture, and those to which attention is particularly called in this article, are the bacteria which act upon nitrogenous matters and oxidise them to nitric acid, or which exert a reducing effect on nitric acid, bringing it to lower forms of oxidation, or even to free nitrogen. These organisms belong to many different species, and act in very many different ways. The general group to which they belong is known as nitro-bacteria. The classification of these organisms by genera and species would prove of little interest to the readers of this article. In general it may be said that there are three distinct genera, comprising, in the first place, those organisms which form ammonia or carbonate of ammonia from organic nitrogenous compounds, such as albumen; in the second place, the organisms which transform carbonate of ammonia into nitrous acid; and in the third place, those which transform nitrous into nitric acid. Each genus is necessary in the complete transformation of proteid matter into nitric acid, in which latter form alone nitrogen is chiefly available for plant-food.

Production of Ammonia.—The bacteria which are especially active in the formation of ammonia are found constantly in surface soils and in the air and rain-waters. By the activity of these organisms in the decomposition of proteid matter, large quantities of ammonium carbonate are produced. The organic carbon which is present in a compound is acted upon during the oxidation of the proteid, and carbon dioxide and certain organic acids are formed. The organic sulphur which is present is converted into sulphuric acid, and the hydrogen partly into water and partly into ammonia. This oxidation is accomplished by bacteria, and, to a less extent, by moulds and yeasts. The table shown on the screen contains the names of the common soil bacteria which ammonise proteid matters. The column headed "per cent" shows the amount of proteid matter changed into ammonia by the several organisms in twenty days, at a temperature of 30°. Of all the bacteria which have been studied, the species *mycoides* has the highest ammonising power, being capable of changing nearly half of the pro-

teid into ammoniacal nitrogen in the time named. In soils where the environment does not permit of the development of the nitrifying ferments, the change stops with ammonia. Such conditions are found in the vegetable soils of swamps, which are extremely acid. In such soils ammonia is quite freely produced, while the nitrous and nitric organisms are absent.

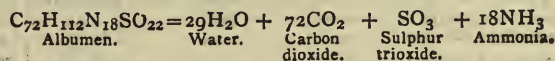
In the analysis of a swamp soil, which had been shown by a bacterial culture to contain no nitrifying ferments, 0.03698 per cent of nitrogen was found as ammonia, and only a trace as nitric acid. In another vegetable soil, which contained nitrifying organisms, 0.0336 per cent of ammoniacal and 0.0474 per cent nitric nitrogen were present. Of the moulds, several have been found capable of producing considerable quantities of ammonia. Among these *Cephalothecium roseum* converted over 30 per cent of proteid into ammoniacal nitrogen in five days, and *Aspergillus terricola* was only a little less active.

The yeasts are still less active, but a large number of them produces ammonia in small quantities.

In general, it may be said that in cultivated soils which have a neutral or alkaline reaction, bacteria are almost the sole ammonia makers, while in vegetable soils of a marked acid reaction, as in swamps and forests, the moulds are the chief producers of ammonia.

In the oxidation of albumen by the *Bacillus mycoides* the carbon is oxidised to carbon dioxide, the sulphur to sulphuric acid, and the hydrogen to water and to ammonia.

The reaction may be expressed by the formula—



The *Bacillus mycoides*, under certain conditions, can form ammonia also from nitrates. In the absence of oxygen it reduces nitrates to ammonia in presence of an organic substance like sugar. In this action it is anaerobic, while in the ordinary process of converting proteid matter into ammonia the action takes place in the absence of oxygen. This is a curious instance of a reverse action produced by the same organism in a different environment, showing, as it does, an oxidising action in the presence of oxygen, and a reducing action in its absence. Some idea of the character of the *Bacillus mycoides*, and the methods of its culture, can be gained by a study of the photographs which will now be projected upon the screen.

Production of Nitrous Acid.—The next step in the process of nitrification is the conversion of ammonia or its compounds into nitrous acid. With a moderate store of ammonia the oxidation into nitrous acid takes place, as a rule, without any of the nitrogen being lost in a free state or being volatilised as ammonia compounds. When, however, there is a large excess of ammonium carbonate, a considerable loss of nitrogen may take place. The practical deduction to be drawn from this fact is apparent. Nitrogenous fertilisers should be applied only in moderate quantities, so as not to increase the stock of material beyond the power of the active ferments to handle it.

The nitrous ferment is by far the largest and most vigorous of the nitrifying organisms. It is from three to four times as large as the nitric ferment, and under a high power of the microscope appears as minute globules, slightly oblate. These globules are multiplied by fission, and the divided parts develop rapidly to perfect organisms of full size. In most cases the organisms appear as distinct globules, but many are congregated into masses where the distinctive cell structure seems to be lost. (A photograph of the nitrous organism was shown upon the screen).

Conversion of Nitrous into Nitric Acid.—The last step in the process of nitrification consists in the oxidation of nitrous to nitric acid. As a rule, plants absorb nitro-

genous food only as nitric acid, but it cannot be said that the nitrogen may not be used by the plant in other forms. Some experiments seem to show that ammonia and its compounds and humus may be directly absorbed by plants, but if this be true it must be only in very limited quantities. The final step, therefore, in nitrification is necessary to secure this valuable food in its most highly available state. The nitrifying organisms are much smaller than their nitrous cousins, and of the same general shape, but more globular.

It must not be supposed that these steps in the preparation of a nitrogenous food are performed with entire distinctness. The impression might be obtained that the ammoniacal ferment exerted its activity, converting the whole of the nitrogenous supply into ammonia, and that in this state only the nitrous ferment would become active and convert the whole product into nitrous acid, which finally, under the influence of the nitric ferment, would form nitric acid. In point of fact, however, in arable soils and under favourable conditions, the steps of nitrification may be almost synchronous. In the case of a growing crop, a chemical examination or repeated chemical examinations might find only traces of ammonia and nitrous and nitric acids. As each particle of ammonia is formed, it is converted without delay into nitrous acid, and then at once into nitric acid. The nitric acid formed is absorbed by the growing plant, and thus it might seem that the activity of the ferments present in the soil had been reduced to a minimum, when in point of fact they were exercising their functions with maximum vigour. The separate stages of nitrification mentioned above can only be secured in the laboratory by a skilled bacteriologist patiently working to separate the different genera of nitrifying organisms until he procures them in an absolutely pure form. As may be supposed, this is very difficult to accomplish.

(A photograph of the nitric organism was shown upon the screen).

(To be continued).

RECOVERY OF WASTE PLATINUM CHLORIDE.

By H. W. WILEY.

ALUMINUM turnings, freed of oil, have been used in this laboratory for some time for many purposes. Immediately after the publication of the paper of Wislicenus and Kaufmann (*Ber. d. Chem. Ges.*, xxviii., 1323) on the various applications of aluminum amalgam in the laboratory, a large quantity of these turnings was procured from the Pittsburg Reduction Co. Considerable difficulty was encountered in attempting to use these turnings in the manner described in the manner cited above. Mr. McElroy prepared the amalgam by washing aluminum clippings with ether to remove oil, treating with dilute caustic soda till free evolution of gas took place, and then washing with water to remove the alkali. The solution of corrosive sublimate was made in alcohol (chosen because the most convenient solvent), diluted with water, and poured over the aluminum. When the evolution of gas was seen to take place from every piece of aluminum in sight, the mercuric chloride solution was decanted and the aluminum washed chloride-free with water. The treatment with soda and mercuric chloride was then repeated. Finally, the turnings were washed free of water with strong alcohol. The washed amalgam in fresh portions of "absolute" alcohol kept up a steady evolution of gas, long after the time all water should have been removed. A portion was removed from the alcohol, washed with ether, and placed in petroleum ether, where the evolution of gas became quite strong. The containing flask was loosely stoppered and stood aside over night. In the morning the petroleum ether was gone.

A fresh portion of amalgam from 200 grms. of aluminum was prepared and treated as before, except that the washing with alcohol was more thorough. The alcohol was removed with ether, and the amalgam finally washed with kerosene. It was then covered with kerosene and stood aside. In about half an hour the evolution of gas became quite violent and the containing bottle hot. On cooling under the tap the generation of gas slackened, but on standing increased again as the mixture warmed up.

The kerosene was such as is used for lamps. It gave a black zone of lead sulphide when treated with the lead acetate test. It is very likely Lima oil. As is well known, in the Frasch process of purifying, the oil is passed through copper oxide, which it converts into copper sulphide. As for every atom of sulphur removed an atom of oxygen must go into the oil, probably the action of the aluminum consisted in appropriating this oxygen. Neither bright sodium or sodium amalgam had any special action on the kerosene used. The action of the amalgam on strong alcohol has been confirmed by Hillyer (*Am. Chem. Journ.*, xviii., 621). We have been able to use these turnings, amalgamated with mercuric chloride, for the reduction of nitrates to ammonia for analytical and other purposes, and it is probable that a speedy and accurate analytical process may be elaborated on this line. We have, however, found the most successful use of the turnings in the recovery of platinum waste. This method of recovery is due to Mr. K. P. McElroy, and has been worked out by him in detail, and has been successfully used for some time in the recovery of platinum chloride waste from potash analyses. The method is as follows:—

The waste platinum from potash determinations is collected and to the hot water solution of platinum potassium chloride is added aluminum in the form of clippings or turnings. In a few minutes a platinum-aluminum couple is formed and reduction goes on vigorously. The addition of hydrochloric acid is not necessary, but is advisable for promoting the settling of the platinum formed. After the reduction is complete, more hydrochloric acid is added to dissolve the excess of aluminum. When this is done the platinum will be found to settle, and the supernatant liquid will be clear. The supernatant liquid contains but little suspended platinum, but it is passed through a large folded filter. If it does not come through clear, as is sometimes the case, return it a few times. As but little platinum gets on the filter, the same filter is used over and over again for successive filtrations, until enough platinum accumulates to make its recovery worth while. When the clear liquid is all decanted, add water to the spongy platinum, shake, allow to settle, and decant. Repeat this until the supernatant liquid is free of chloride. The spongy platinum is then covered with strong nitric acid and heated for the purpose of removing copper. Aluminum often contains a little copper, which of course remains with the platinum. When the copper is all dissolved, the copper nitrate and the excess of nitric acid are removed by washing with water by decantation as before, till the supernatant liquid is acid free when tested with Congo paper. The resulting platinum black is dissolved with aqua regia, made by mixing five parts of hydrochloric acid with one part of nitric, added in amount sufficient to dissolve all of the platinum present. The solution thus obtained is transferred to a porcelain dish and evaporated on a steam-bath till a portion taken out with a rod solidifies on cooling. The residue is diluted with water and hydrochloric acid and re-evaporated. If, on adding water to the syrupy mass formed by this evaporation, nitrous vapours are evolved, add plenty of water and re-evaporate. Repeat this evaporation with water till the nitrous vapours are no longer evolved on dilution. Finally, dilute sufficiently to filter and add water until the colour of a platinum chloride solution of known content is matched.—*Journal of the American Chemical Society*, xix., No. 3.

NOTICES OF BOOKS.

The Electric Furnace. ("Le Four Électrique"). By HENRI MOISSAN, Membre de l'Institut. Paris: G. Steinheil. 1897. Pp. 385.

(FIRST NOTICE).

M. MOISSAN was led, he tells us, to the invention of the electric furnace by the necessity of having an extremely high temperature to enable him to carry on his experiments on the crystallisation of carbon from its solution in molten iron; this can be achieved at about 1000°, but as he aimed at working on fairly large quantities of material, he found the ordinary methods employed inadequate.

The book is divided into four chapters. The first deals with the different patterns of furnaces used, and their application to the study of the fusion and volatilisation of a certain number of refractory bodies. Chapter II. comprises the study of three varieties of carbon, viz., amorphous carbon, graphite, and diamond. In Chapter III. we have the preparation of several *simple* bodies by means of the electric furnace, and the experiments carried out on chromium, manganese, molybdenum, tungsten, uranium, vanadium, zirconium, titanium, silicon, and aluminium; while in the fourth and last chapter we find described the research on a new series of *binary* compounds, such as carbides, silicides, and borides. The preparation of carbide of calcium, in particular, has been the subject of a distinct research, and is described in detail.

The highest temperatures hitherto attained industrially range between 1700° and 1800°. The invention of the oxy-hydrogen blowpipe by MM. Sainte-Claire Deville and Debray rendered great services to chemistry, but the highest temperature obtained by its means was not more than 2000°. Other workers, before M. Moissan, had used the intense heat developed by the electric arc, notably Despretz, Siemens, Huntingdon, Cowles, and others; but there were objections to all their furnaces, though they were suitable to the purposes for which they were devised.

The author of this work insists that his furnace is not an industrial one, but is meant solely for research; his primary object being to concentrate as much heat as possible into the smallest possible space. His early experiments were carried out by means of a small Gramme dynamo, giving a current of 35 to 40 ampères at a pressure of 55 volts. But as the work proceeded, this was found to be insufficient, and by the courtesy of the directors of some of the large electric lighting companies, M. Moissan was enabled to avail himself of much more powerful currents; on a few occasions using as much as 300 h.p. at one time.

M. Moissan's first furnace was made of quicklime, and was exhibited at the Académie des Sciences in December, 1892. It consisted of two well-dressed bricks of lime placed one on the other, the lower one containing a small cavity which served as a crucible, with two grooves from it to the outside, to hold the electrodes. The upper brick was slightly hollowed out over the cavity in the lower one, and as the surface is soon melted it becomes, so to say, polished, and acts as a reflector, increasing the heat in the cavity below. The cavity could also be used to hold a small carbon crucible containing the substance to be melted. The great point of difference between this furnace and all previous models is that, in this new one, the material under treatment does not come in contact with the electric arc—that is to say, with the vapour of carbon. Another great convenience is that the electrodes are movable, thus affording great facilities for striking and regulating the arc. The dimensions of the bricks first used were as follows:—The upper, 18 c.m. × 15 c.m. × 5 c.m. thick; the lower, 18 c.m. × 15 c.m. × 8 c.m. thick. This was large enough for working with a current of 100 to 125 ampères and 50 or 60 volts; by increasing

the length to 22 c.m. or 25 c.m., one can use a current of 450 ampères and 75 volts.

Very great care had to be exercised in the manufacture of the electrodes, so as to, as far as possible, avoid all chance of impurities.

To demonstrate the facility with which quicklime can be volatilised, no crucible need be employed, the bricks themselves supplying the material for the experiment. As soon as the arc is established there is a strong smell of hydrocyanic acid, the small quantity of water remaining in the electrodes forming, with the carbon, acetylene; this gas in the presence of nitrogen, under the powerful action of the arc, accomplishing the synthesis of hydrocyanic acid, as discovered by Berthelot. This, however, does not continue long. The regulation of the arc must be carefully attended to, as the furnace is cold at first, but, on warming, the carbons can be separated until they are 2 c.m. or 2½ c.m. apart. With a current of 400 ampères and 80 volts, the flames issuing from the grooves holding the electrodes are accompanied, after five or six minutes, by torrents of white smoke, produced by the volatilisation of the lime, and this can easily be condensed on any cold surface. With a current of 800 ampères and 110 volts more than 100 grms. of lime can be volatilised in five minutes; this bears testimony to the enormous power of the furnace. On removing the cover at the end of the experiment, it can be seen that the lime in the cavity is actually melted, and by allowing the cover to cool gradually, veritable stalactites are formed.

For some experiments, carbonate of lime is preferable for making the body of the furnace; it possesses greater solidity or compactness than quicklime, and is easily obtained in large blocks.

When using such enormous currents as 1200 to 2000 ampères and 100 volts, lime bricks are speedily put *hors de combat*, and proper manipulation becomes out of the question; in such a case the cavity in the brick is enlarged and lined with alternate plates of magnesia and carbon, the magnesia being next to the lime, and the carbon on the inside. By such an arrangement the carbon and lime, which would otherwise form carbides of lime, are kept apart, and as magnesia is not reducible by carbon, the furnace remains uninjured.

Another modification of the electric furnace consists in inserting a pair of carbon tubes through the lower brick in such a manner that they are at right angles to the electrodes and about 1 c.m. below the arc; by their means the gases inevitably driven off by the intense heat of the arc find a ready exit, and the experiment can be continued for several hours instead of minutes. The tubes and the electrodes are of course kept from contact with the lime or chalk by means of a protective layer of magnesia, and it is noticed that the ends of the carbon tubes exposed to this extreme temperature are entirely converted into graphite. By inclining the tube to an angle of 30° the furnace can be employed to produce the most refractory metals in quantity; thus in an hour 2 kilogrammes of chromium were reduced from oxide and run into an ingot.

A further modification, or rather extension, of the furnace consists in arranging several arcs in parallel; by this means a greater quantity of heat can be obtained and large quantities of metals produced without necessarily going to a great extreme of temperature; but, if required, a large and continuous stream of highly refractory metal can be obtained at a melting-point of considerably over 3000°. M. Violle has put the highest attainable temperature at 3500°.

Journal of Agriculture, published by the Department of Agriculture, Cape of Good Hope. March 18, 1897. Cape Town: Townshend, Taylor, and Snashall.

No. 6 of this journal has just reached us; it contains several interesting articles on subjects connected with agricultural and farming work, fruit growing, forestry, &c.

Some interesting remarks are to be found in a short article entitled "Bacteriology and the Plague," in which reference is made also to the work of certain French savants in the Transvaal who are engaged in the study of the rinderpest.

The great difficulty to be contended with in serious epidemics of this kind, is to provide at short notice, and keep up an efficient supply of anti-toxic serum. Its preparation is naturally a long and cautious process, as each individual animal can only yield at intervals a limited quantity of serum without feeling ill effects. Long before the present epidemic, the bacteriological treatment of disease had been developed by the Government of India on a much greater scale than that attempted by any European Government. A year and a half ago, in the Presidency of Bengal alone, 42,445 persons were inoculated for cholera, without a single mishap, or injury to health.

Traité Élémentaire de Chimie à l'Usage des Candidats au Certificat d'Aptitude des Sciences Physiques, et Naturelles et des Candidats aux Baccalauréats Scientifiques. Paris: Georges Carré and C. Naud. 1896.

Chimie Minérale. Par A. HALLER, Correspondant de l'Institut, Directeur de l'Institut Chimique de Nancy, and P. TH. MÜLLER, Maître de Conférence à l'Institut Chimique de Nancy. Pp. 336.

Chimie Organique. (By the same Authors).

THE work on Mineral Chemistry is a good and useful exposition of chemical principles and the elementary laws of chemical science. There is a catalogue of the elements in which argon and helium duly figure, but neodymium and praseodymium are not recognised as separate elements. The laws of Dalton, of Gay-Lussac, the hypothesis of Avogadro and Ampère, are fully expounded; also the determination of molecular and atomic weights are given, followed by an outline of crystallography.

The principles of thermo-chemistry are introduced after the halogens.

Oxygen, sulphur, selenium, and tellurium are made to rank as the second family of the "metalloids." For the recognition of ozone the authors recommend the use of thallium paper in the first place, but Hurter's reagent is not omitted. Mineral waters are here divided into eight classes—the acidulous, the alkaline, the sulphuretted, the chlorides, bromides, and iodides, the sulphate, the iron waters, and the arsenical.

The descriptions of the several elements are followed by a sketch of the periodic classification, here ascribed solely to Mendeleeff. An abstract of qualitative analysis concludes the work.

The authors have accomplished their task ably as far as its limited scope would permit.

In the work on Organic Chemistry, the authors, in their introductory remarks, draw the accepted distinction between organic compounds and organised bodies. They classify organic matter as containing two elements only, such as benzene, or three elements, such as alcohol, or acetic acid, or, again, four elements, as is the case with urea and indigo. As a supplement follow artificial compounds into which a number of extraneous elements have been introduced.

At the end of the work we find an account of products of unknown constitution, such as biliary products, gelatins, albumenoids, syntonines, peptones, enzymes, milk, blood, blood-pigments, &c. We do not find any special mention of the "toxines."

Within its scope this work fulfils the reasonable expectation of the student.

The Principles of Mathematical Chemistry. The Energetics of Chemical Phenomena. By Dr. GEORG HELM, Professor in the Royal Technical High School, Dresden. Authorised Translation from the German, by J. LIVINGSTON R. MORGAN, Ph.D. (Leipzig), Instructor in The Brooklyn Polytechnic Institute. New York: John Wiley and Sons. London: Chapman and Hall, Lim. 1897. Pp. viii.—228, 12mo., cloth.

DR. HELM's well-known "Grundzüge der mathematischen Chemie," published in 1894, is now offered to students in an English dress.

The translation follows the text as closely as possible, and Dr. Morgan has "aimed at clearness rather than literary style;" yet the involved sentences peculiar to German only occasionally appear through the translation.

The work is divided into four parts. Part I. dealing with the measurement of chemical energy, mechanical energy, and the volume energy of gases. Part II. is devoted to entropy, and discusses the thermodynamics of perfect gases, the entropy of gases and gas mixtures, the relations between heat and volume energy, as well as electrical energy. Part III. treats of chemical intensity, concluding with a chapter on the velocity of a chemical reaction. Part IV. considers the degrees of freedom of chemical phenomena, such as the rule and equilibrium of phases.

Students of chemistry who have the habit of dealing with subjects on a mathematical basis will find this volume of great service, throwing much light on the recent developments of the science. It is not a book for beginners, but one which advanced classes should take up with great profit. The author frequently refers to Wilhelm Ostwald's writings, to which Helm's book forms a good guide.

The labours of Willard Gibbs in this direction are more than once gracefully acknowledged by the author. An index closes the volume. H. C. B.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 16, April 20, 1897.

Determination of the Surface, the Bulk, and the Chemical Composition of the Human Body.—Ch. Bouchard.—The disassimilation is proportional to the weight of the body, or rather to the weight of fixed albumen, and the functional or respiratory consumption is proportional to the surface of the body. The products of nutrition, i.e., the chemical products (urea, carbonic acid, and water), and the dynamic products (calories) depend on the quantity of fixed albumen and the intensity of its activity.

Details of the Method followed in precise Cryoscopic Researches.—F. N. Raoult.—The author has succeeded in measuring the reductions of the freezing point with an approximation of 0.0065°. By this means he has made a methodical study of the influence of superfusion upon the congelation-point of aqueous solution.

On the Physiological Action of the X Rays.—W. Crookes.—I can entirely corroborate the observations of the learned author of the paper just read (memoir by M. Sorel, *Comptes Rendus*, Session of April 12, p. 826). I believe, nevertheless, that the X rays act upon different persons with a different intensity. In particular, I have worked for a long time with tubes producing rays of this kind, and I have perhaps been exposed to their action for a longer time than most experimentalists, but without

undergoing any effects either upon the face or the hands. On the other hand, I have observed very marked physiological effects, analogous to those which have been just spoken of, produced upon persons who had been exposed to the X rays. I am therefore inclined to think that the very energetic action sometimes observed with these rays depends, up to a certain point, on the idiosyncrasy of the experimentalist.

Comparison of the Absorption by Crystallised Media, of Luminous Rays, and X Rays.—V. Agafonoff. —On studying my proofs I was surprised to find that there exists a general opposition between the absorption for the luminous rays and for the Röntgen rays. The sulphates very transparent for the ultra-violet rays are extremely opaque for the X rays. The inverse holds good for the majority of crystalline organic compounds. The nitrates absorb the luminous rays more than the sulphates and less than organic bodies; the X rays, on the contrary, less than the sulphates and more than organic bodies.

Black Light.—M. Perrigot.—Black light plays no part in the explanation of phenomena connected with facts the law of which is perfectly known.

Separation of Chlorine and Bromine.—H. Baubigny and P. Rivals.

Separation of Nickel from Cobalt and Iron, and of Cobalt from Aluminium.—E. Piñerua.—(See p. 193).

On Cholesterine.—Ch. Cloëz.—Cholesterine can combine with a single atom of bromine to form a body, $C_{26}H_{44}OBr$, less soluble in cold carbon disulphide than is cholesterine itself and its dibromide.

MISCELLANEOUS.

Royal Institution.—The Annual Meeting of the Members of the Royal Institution of Great Britain was held on Saturday afternoon, May 1st, at the house of the Institution in Albemarle Street, Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, presiding. The Annual Report of the Committee of Visitors for the year 1896, testifying to the continued prosperity and efficient management of the Institution, was read and adopted. Fifty-eight new Members were elected in 1896. Sixty-four Lectures and nineteen Evening Discourses were delivered in 1896. The books and pamphlets presented in 1896 amounted to about 274 volumes, making, with 621 volumes (including periodicals bound) purchased by the Managers, a total of 895 volumes added to the Library in the year. Thanks were voted to the President, Treasurer, and the Honorary Secretary, to the Committees of Managers and Visitors, and to the Professors, for their valuable services to the Institution during the past year. The following gentlemen were unanimously elected as Officers for the ensuing year:—

President—The Duke of Northumberland, K.G., D.C.L., LL.D.

Treasurer—Sir James Crichton-Browne, M.D., LL.D., F.R.S.

Secretary—Sir Frederick Bramwell, Bart., D.C.L., LL.D., F.R.S., M. Inst. C.E.

Managers—Sir Frederick Abel, Bart., K.C.B., D.C.L., LL.D., F.R.S.; The Right Hon. Arthur James Balfour, M.P., D.C.L., LL.D., F.R.S.; John Wolfe Barry, Esq., C.B., F.R.S., M. Inst. C.E.; William Crookes, Esq., F.R.S.; Edward Frankland, Esq., D.C.L., LL.D., F.R.S.; Charles Hawksley, Esq., M. Inst. C.E.; Donald William Charles Hood, M.D., F.R.C.P.; Victor Horsley, Esq., M.B., F.R.S., F.R.C.S.; William Huggins, Esq., D.C.L., LL.D.,

F.R.S.; The Right Hon. Lord Lister, M.D., D.C.L., LL.D., Pres. R.S.; Ludwig Mond, Esq., Ph.D., F.R.S.; Arthur William Rucker, Esq., M.A., D.Sc., F.R.S.; Basil Woodd Smith, Esq., F.R.A.S., F.S.A.; The Hon. Sir James Stirling, M.A., LL.D.; Sir Henry Thompson, F.R.C.S., F.R.A.S.

Visitors—Sir James Blyth, Bart.; William Arthur Brailey, M.D., M.R.C.S.; Edward Dent, Esq.; John Ambrose Fleming, Esq., M.A., D.Sc., F.R.S.; Edward Kraftmeier, Esq.; Sir Francis Laking, M.D.; Hugh Leonard, Esq., M. Inst. C.E.; Sir Philip Magnus, J.P.; T. Lambert Mears, Esq., M.A., LL.D.; Lachlan Mackintosh Rate, Esq., M.A.; Thomas Tyrer, Esq., F.C.S., F.I.C.; Roger William Wallace, Esq., Q.C.; John Westlake, Esq., Q.C., LL.D.; His Honour Judge Frederick Meadows White, Q.C.; James Wimshurst, Esq.

University of London.—The following Examiners for the year 1897-8 were elected at the Meeting of the Senate, held April 28th, 1897:—Mathematics and Natural Philosophy—E. W. Hobson, Sc.D., F.R.S., and Joseph Larmor, D.Sc., M.A., F.R.S. Experimental Philosophy—Prof. G. F. FitzGerald, M.A., F.R.S., and Prof. Silvanus Thompson, D.Sc., B.A., F.R.S. Chemistry—Prof. Wyndham R. Dunstan, M.A., F.R.S., and Prof. William Ramsay, Ph.D., F.R.S. Geology and Physical Geography—Prof. T. G. Bonney, Sc.D., LL.D., M.A., F.R.S., and Prof. Charles Lapworth, LL.D., F.R.S. Materia Medica and Pharmaceutical Chemistry—Sidney Phillips, M.D., and W. Hale White, M.D. Forensic Medicine—Prof. J. Dixon Mann, M.D., and Thomas Stevenson, M.D.

Speed of Esterification, as compared with Theory. Robert B. Warder (*J. Phys. Chem.*, i, 149).—The author shows that the rate of esterification of alcohol and the three chloracetic acids, as determined by Lichty (*Tech. Quart.*, viii, 99), does not conform to the requirements of the laws of mass-action in the form applicable to a reversible reaction of the second order, and he suggests four possible causes of the deviations.—*Journ. Amer. Chem. Soc.*

On the Volatility of Ferric Chloride.—Henry P. Talbot (*Am. Chem. J.*, xix, 52-59).—The experimental data show that no loss of ferric chloride occurs, when its solutions (whether neutral or acidified with hydrochloric acid) are evaporated to dryness on the water-bath or upon the hot plate, provided in the latter case they are not too strongly overheated. The residues so obtained were subjected to the temperature usually employed to dehydrate silicic acid (130°C) for two hours, but suffered no loss of iron. Prolonged heating of these residues over a free flame occasioned but a slight loss (0.4 per cent) of the iron present. Concentrated acid solutions of the chloride, when boiled in a distilling flask, allowed ferric chloride to pass into the receiver only when a slight separation of the solid had taken place on the side of the flask, which, in the acid atmosphere, was volatilised by the overheating of the glass. When ferric chloride solutions are evaporated with exposure to the air, a loss of chlorine ensues, and the basic ferric salt formed prevents loss of the iron as chloride. The presence of ammonium chloride with the ferric chloride occasioned no loss of the latter, even at 130°C . The residues, when heated over a free flame, suffered a loss of iron, as would be expected. The presence of aqua regia with the ferric chloride solution tends to occasion a slight loss of iron during evaporation. The maximum loss was 0.6 per cent of the iron present, but in other cases very little or no loss could be detected. Vogel's experiments (*N. Rep. Pharm.*, xviii, 157) were repeated, and it was found that a slight volatilisation of iron seems to take place from an ethereal solution at the temperatures of the laboratory, but, on the other hand, the presence of ether or its vapour does not promote the volatilisation of the ferric chloride from its boiling, concentrated, aqueous solutions.—*Journ. Amer. Chemical Soc.*

Pottery and Glass Trades Benevolent Institution.

—By way of celebrating the Diamond Jubilee of the reign of Queen Victoria, and of benefitting the funds of this Institution, entertainments have been organised by the Board of Management. On Wednesday, May 12th, there will be a *Conversazione and Dance* at the Galleries of the Royal Institute of Painters in Water Colours, Prince's Hall, Piccadilly, on which occasion Sir Henry Doulton and Miss Doulton will receive the guests. On Wednesday, July 7th, a Dinner will be held in the Prince's Hall, Hotel Cecil, Salisbury Street, Strand, in aid of the Diamond Jubilee Celebration Fund, when William Woodall, Esq., M.P., for Hanley, will preside. The Board will be pleased to receive subscriptions, and the names of ladies or gentlemen who will kindly consent to act as Stewards. Tickets and further information may be obtained from the Secretary, Mr. A. J. Prickett, 6, Thavies Inn, Holborn Circus, E.C.

The Imperial Hygienic Laboratories of Japan.—Soon after Japan had, in 1869-70, made the treaties now in force with foreign countries, medicines were imported in large quantities, and, in order to protect the public against quackery, the Department of Education established a sub-department or Medical Bureau to examine and report on the quality of all medicines imported. This department has gradually grown, and so increased its scope that it now undertakes the analysis and examination of all kinds and sorts of substances. During the year 1895 the total number of bottles, cans, bags, and other packages examined, amounted to 1,122,733, of which 63,277 were reported to be unfit for use: these figures give an idea of the enormous amount of work done, and it is interesting to note that for some years past the work has been entirely done by the Japanese themselves.

Sixty-ninth Meeting of the German Society of Science and Arts at Brunswick, Sept. 20th to 25th, 1897.—At the meeting of this Society in September next there will be a sitting devoted to the question of the constitution of camphor, and it is requested that any papers or notes which any members of the profession desire to contribute may be sent in by the middle of May, so that they can be included in the official programme which is to be issued at the beginning of July. It is intended to devote Wednesday, the 22nd of September, to a general meeting of all who are interested in the subject of Photography as applied to scientific investigations; and Prof. H. W. Vogel, of Charlottenburg, has promised to deliver the introductory address. There will also be an exhibition of scientific photographs organised by Prof. Max Müller; contributions of papers and photographs are requested from all workers on the subject.

MEETINGS FOR THE WEEK.

- MONDAY, 10th.—Society of Arts, 8. (Cantor Lectures). "Design in Lettering," by Lewis Foreman Day.
TUESDAY, 11th.—Royal Institution, 3. "Volcanoes," by Dr. Templest Anderson, B.Sc.
— Society of Arts, 8. "A Half Century of Line Engraving, 1780-1830," by George Clulow.
WEDNESDAY, 12th.—Society of Arts, 8. "Motor Traffic—Technical Considerations," by Sir David Salomons.
THURSDAY, 13th.—Royal Institution, 3. "Liquid Air as an Agent of Research," by Prof. Dewar, F.R.S., &c.
FRIDAY, 14th.—Royal Institution, 9. "Explosion—Flames," by Prof. Harold Dixon, F.R.S.
— Physical, 5. "Instrument for Comparing Thermometers with a Standard," by W. Watson. "Experiment in Surface Tension," by A. S. Ackerman. "Effect of Temperature on the Magnetic and Electric Properties of Iron," by D. K. Morris. "Formation of Mercury Films by Electric Osmosis," by Rollo Appleyard.
SATURDAY, 15th.—Royal Institution, 3. "The Greek Theatre according to Recent Discoveries," by the Rev. J. P. Mahaffy, D.D.

Water-Glass, or Soluble Silicates of Soda and Potash, in large or small quantities, and either solid or in solution, at ROBERT RUMNEY'S, Ardwick Chemical Works, Manchester.

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1955.

ON THE FERGUSONITE METALS.

I. ON PHILIPPUM.

By M. MARC DELAFONTAINE.

AFTER long-continued work on the Gadolinite and Samarskite earths, I came to the conclusion—published in 1878 and 1880—that the yellow oxide erbia discovered by Mosander in 1843 contained two yellow earths, which I called *terbia* and *philippia*. Later on the individuality of the latter was denied by two English chemists. But M. Marignac mentioned it at the close of his elaborate paper on the Samarskite Earths (May, 1880), and Mr. W. Crookes, who first rejected it, mentions it among some of his fractional products of yttria.

The lack of time and insufficient means have long prevented my completing the work, by which I expected to justify my conclusions. The investigation of the Fergusonite earths, though incomplete, enable me to show that the characteristics of philippium are such as to deserve the attention of those who are discussing the Periodic Law and the necessary modifications to M. Mendeleeff's classification of elements.

My results in regard to the other Fergusonite metals will be the subject of a second paper, now nearly finished.

Occurrence.—Philippium has been found in Gadolinites, Samarskite, and the mineral from Bluffton (Slano county, Texas), described and analysed as Fergusonite by MM. Hidden and Mackintosh. I secured several pounds of that Fergusonite from Mr. English, mineralogist, in New York; it has proved to be the best material for the extraction of philippium.

Extraction.—500 grms. of the powdered ore were treated at a time, in a large leaden dish, with three times their weight of strong hydrofluoric acid. The reaction is attended with much heat and a great effervescence,—hence the necessity of having the lead vessel not more than half full of the mixture. When the bubbling has subsided, a moderate heat is applied as long as the stirred mass gives off bubbles. An equal volume of water is added; the acid fluorides dissolve, and the earthy fluorides remain as a sediment, coloured green by uranium. The supernatant fluid is poured out, and several times replaced by water. The insoluble fluorides are then transferred into a large platinum dish, and decomposed by sulphuric acid. The solution of the resulting sulphates containing uranium in the uranous form, it is then time to oxidise the latter with some hydrogen peroxide, or, better, some sodium dioxide, which at once turns the liquid yellow.

Oxalic acid throws down the earthy oxalates; iron and uranium remain dissolved. After ignition of the oxalates the earths are left as a moderately dark yellow powder, completely and readily soluble in nitric acid. The mixed salts show an absorption spectrum, the lack of intensity of which indicates a very small proportion of didymium, and not much more of erbium. The pink colour of the solution is, however, much stronger than we might expect from so small a proportion of the absorbing nitrates. The same fact was noticed before by M. Marignac and myself on other occasions.

Philippium may be separated from that mixture of nitrates by different methods, all based upon the fact that its basic energy is less than that of its congeners. Fractional precipitations, by dilute ammonia or by potassium bioxalate, have been resorted to. I give preference to the fractional decomposition of the nitrates by heat. In that

way philippium is separated first, then a pale-coloured earth without an absorption spectrum, followed by the erbia earths, and finally terbia and yttria.

The mass of nitrates is heated in a flat-bottomed dish; it foams much for a while, then comes to a quiet fusion and turns red. After some minutes of that quiet decomposition, the dish is allowed to cool. The solid residue is a beautiful orange-red glass, which water dissolves in part only, leaving a dark yellow gelatinous basic salt.

The same process is repeated on the parts taken up by water as long as a coloured residue is left. It sometimes happens that the glass-like mass re-dissolves entirely in water, and makes an opalescent or milky yellowish liquid with a decided greenish fluorescence. That may be corrected by the addition of dilute ammonia, followed by a digestion of several hours in a warm place; I find it preferable to evaporate the whole liquid again, and decompose it at a somewhat higher temperature. The subsalts thus obtained were sorted according to their colour, and subjected to new series of decompositions, until a bright orange-yellow basic nitrate was obtained, which dissolved slowly in moderately dilute nitric acid, and thus gave a deep orange-red transparent solution. Sometimes the solution is entirely colourless, at once.

Characteristics.—There are two series of philippium compounds—the philippous and the philippic ones, corresponding to a white and to an orange oxide. The salts of the first series are colourless, quite stable, and generally crystallise well; they correspond to the lanthanum and yttrium salts. Their solutions do not seem to give an absorption-spectrum; truly they sometimes faintly show the erbium lines and bands under a thickness of 5 or 6 c.m.; but that is evidently due to a very small proportion of erbium metals, which it is very hard to get rid of entirely.

Potassium-philippous sulphate is soluble in a saturated solution of potassic sulphate. The *formiate* crystallises very slowly from a syrupy solution, in fibro-radiated masses. By ignition, the *oxalate* dried at 130° C. yields about 51.5 per cent of orange-coloured oxide. The crystallised *nitrate* is colourless; when heated it melts, and decomposes into a reddish glass, not entirely soluble in water, but very apt to make a colloidal solution which passes very slowly through the filter.

Philippic oxide has a deep orange-red colour, most intense in the oxide made from the calcination of the nitrate or the acetate. Its moist hydrate is light yellow. When air-dried it is in small and somewhat darker lumps; by calcination it turns dark red. With moderately dilute cold nitric acid, the latter makes a yellow solution; in strong acid it dissolves with effervescence and heat, and gives the colourless philippous nitrate. With hydrochloric acid, philippic oxide evolves chlorine, and gives the proto-salt. Other acids dissolve it by boiling with some alcohol.

Equivalent.—Although philippium shows a very close resemblance to yttrium and cerium, the constitution of its compounds remains to be established by crystallography or otherwise.

SO₃(=80) combines with about 96 of philippous oxide, which makes Pp 80, or 120, or 160, according to whether the oxide is PpO, Pp₂O₃, or even PpO₂.

Relationship to Other Elements.—Philippium is more closely allied to cerium and terbium than to any other of the yttrium and cerium metals. It is to yttrium what cerium is to lanthanum. Its equivalent, the colour of its subnitrates and that of the philippic salts, the solubility of its formiate, separate philippium from terbium. These characteristics, and the solubility of potassium-philippo sulphate in potassium sulphate solutions, distinguish it from the two ceriums of M. Brauner and M. Schützenberger. A heated mixture of cerium nitrate with that of the Fergusonite earths (left after the removal of Pp) does not behave at all like the original nitrates; the residue of cerium subnitrates does not resemble the corresponding compound of philippium. Terbium nitrate melts into a

colourless glass which after partial decomposition is not yellow, and leaves no yellow residue after washing.

Chicago, April 24, 1897.

ON THE SEPARATION OF THORIA FROM ZIRCONIA.

By M. MARC DELAFONTAINE.

MR. GLAZER'S paper on the Separation of Thoria and Zirconia, in recent numbers of the *CHEMICAL NEWS*, prompts me to describe here the method which I have applied for years to similar cases, with accurate results. The fusion of the mineral with potassium bisulphate does not work well in the case of mixtures containing titanous acid with zirconia. I proceed as follows:—

The powdered mixture (ore or oxides) is fused in a platinum crucible with twice its weight of acid potassium fluoride (KHF_2). The zirconia is separated as potassium fluozirconate, K_2ZrF_6 , from the solidified mass by means of boiling water containing a few drops of HF .

The insoluble fluorides decomposed by sulphuric acid, and ignited below a dull red heat, will leave thorium, cerium, and other earths, as sulphates. Silica, if present, escapes as silicon fluoride. The sulphates are dissolved in water and precipitated by oxalic acid, the oxalates being treated by a saturated hot solution of ammonium oxalate; the thorium salt is dissolved and cerium left. The ignited oxalates left the oxides in good condition for further work. Zirconia is thrown down from its fluo-salt by ammonium hydrate.

Titanium, if present in the original mixture, is found as fluotitanate soluble in hot water. Hydrogen peroxide would separate it from the zirconia.

Chicago, April 24, 1897.

SOIL FERMENTS IMPORTANT IN AGRICULTURE.*

By HARVEY W. WILEY,

Chief of the Division of Chemistry, Department of Agriculture,
Washington, D.C.

(Concluded from p. 224).

Ferments Oxidising Free Nitrogen.—In the preceding paragraphs the attention of the reader has been briefly called to the action of those species of ferments which attack nitrogen in some of its forms of combination. Since nitrogenous food is the most expensive form of nutriment which the plant consumes, it is a matter of grave importance to agriculture to know the full extent of the supply of this costly substance. It is evident that the continued action of nitrifying ferments finally tends to exhaust the stores of this substance which have been provided in the soil. The quantities of oxidised nitrogen produced by electric discharges in the air and by other meteorological phenomena, and which are brought to the soil in rain waters, are of considerable magnitude, but lack much of supplying the ordinary wastage to which the stores of soil nitrogen are subjected. Even with the happiest combination of circumstances, it is not difficult to see in what way the available stores of nitrogen could be diminished to a point threatening the proper sustenance of plants, and thus diminishing the necessary supplies of human food. The examination of the drainage waters which come from a fertile field in full cultivation, is sufficient to convince the most sceptical of the fact that the growing crop does not by any means absorb all of the products of the activity of the nitrifying ferments. Nitric acid and its compounds,

the nitrates, are exceedingly soluble in water, and for this reason any unappropriated stores of them in the soil are easily removed by heavy downpours of rain. Happily the living vegetable organism has the property of withholding nitric acid from solution, either by some property of its tissues or more probably by some preliminary combination which the nitric acid undergoes in the plant itself. This is easily shown by a simple experiment. If fresh and still living plants be subjected to the solvent action of water, very little nitric acid will be found to pass into solution. If, however, the plants be killed before the experiment is made, by being exposed for some time in an atmosphere of chloroform, the nitric acid which they contain is easily extracted by water.

The losses, therefore, which an arable soil sustains in its content of nitrogenous matter must be supplied either by the addition of nitrogenous fertilisers or by some action of the soil whereby the nitrogen which pervades it may be oxidised and fixed in a form suited to the nourishment of plants. The discussion in regard to the possibility of fixing nitrogen in the soil has been carried on with great vigour during the last two decades. The proof, however, is now overwhelming that such fixation does take place. It would not be proper here to enter into a discussion of the processes by which this fixation is determined, and, in fact, they are not definitely known. One thing, however, is certain, viz., that it is accomplished by means of micro-organisms or ferments similar, perhaps, in their nature to those already mentioned, but capable of absorbing, assimilating, and oxidising free nitrogen.

Methods of Oxidising Free Nitrogen.—At the present time it is sufficiently well known that this operation takes place in two ways. In the first place, there are found to exist on the rootlets of certain plants, chiefly of the leguminous family, colonies of bacteria whose function is known by the effects which they produce. In such plants in a state of maturity, as was mentioned above, are found larger quantities of organic nitrogen than could possibly have been derived from the soil in which they were grown or from the fertilisers with which they were supplied. Cultural experiments in sterilised soils, with careful exclusion of all sources of organic nitrogen, have proved beyond question that this gain in nitrogen is found only in such plants as are infected by the organism mentioned. The logical conclusion is therefore inevitable that these organisms, in their symbiotic development with the plant rootlets, assimilate and oxidise the free nitrogen of the air and present it to the plant in a form suited to absorption. Attempts have been made to inoculate the rootlets of other families of plants with these organisms, but so far without any pronounced success. There are, however, certain orders of low vegetable life, such as cryptogams, for instance, which seem to share to a certain degree the faculty of the leguminous plants in acting as a host for the nitrifying organisms mentioned. The observation above recorded becomes a sufficient explanation of the fact that the fertility of fields is increased by the cultivation of leguminous plants, which would not be possible except they possess some such property as that which has already been described.

Another order of organisms has also been discovered which is capable of oxidising free nitrogen when cultivated in an environment from which organic nitrogen is rigidly excluded. It seems probable, therefore, even in soils which bear crops not capable of developing nitrifying organisms on their rootlets, that the actual stores of available nitrogen may be increased. This fact explains the observation which has frequently been made that in fields which are not cultivated, but which remain in grass, there may be found an actual increase in the total amount of nitrogen which is available for plant growth. As will be seen further along, the soil is also infested with an organism which is capable of destroying nitric acid and returning the nitrogen which it contains to the air in a free state. It seems almost certain that in every complete decomposition of a nitrogenous organism a part of the

* Abstract of a Lecture delivered before the Chemical Section of the Franklin Institute, February 16th, 1897.

nitrogen which it contains escapes in the free state. Were it not, therefore, for the fact that this free nitrogen can be again oxidised and made available for plant growth, the total stores of organic nitrogen in existence would be gradually diminished, and the time would ultimately come when their total amount would not be sufficient to sustain a plant life abundant enough to supply the food of the animal kingdom. Thus the Earth itself, even without becoming too cold for the existence of the life which is now found upon it, might reach a state when plant and animal life would become practically impossible by reason of the deficit of nitrogenous foods.

Much less is known concerning the character and activity of the organisms that oxidise free nitrogen than of those which feed upon organic nitrogen. It cannot be doubted, however, that these scarcely known ferments are of the greatest importance to agriculture, and the further study of their nature and the proper methods of increasing their activity cannot fail to result in the greatest advantage to the practical farmer. (Photographs showing the occurrence of the nitrifying tubercles of leguminous and other plants were shown upon the screen).

Fertilising Ferments.—Two years ago I used the following words in a Report published by the Department of Agriculture:—

"When a soil is practically free from albumenoid bodies, and contains but little humus, the attempt to develop a more vigorous nitrifying ferment would be of little utility. Even in a soil containing a considerable degree of humus, it may be found that its nitrogen content has been so far reduced as to leave nothing practically available for the activity of nitrification. In such cases the only rational method of procedure is in the application of fertilisers containing nitrogen. In other cases, where the lack of fertility is due to the extinction or attenuation of the nitrifying ferment, remunerative results may be obtained by some process of seeding similar to that described above. It is entirely within the range of possibility that there may be developed in the laboratory species of nitrifying organisms which are particularly adapted for action on different nitrogenous bodies. For instance, the organism which is found most effective in the oxidation of albumenoid matter may not be well suited to convert amides or the inert nitrogen of humus into nitric acid. We have already seen the day when the butter-maker sends to a laboratory for a ferment best suited to the ripening of his cream. It may not be long until the farmer may apply to his laboratory for particular nitrifying ferments to be applied to such special purposes as are mentioned above. Because of the extreme minuteness of these organisms, the too practical agronomist may laugh at the idea of producing fertility thereby; and this idea, indeed, would be of no value were it not for the wonderful facility of propagation which an organism of this kind has when exposed to a favourable environment." It is true that the pure cultures which the laboratory would afford would be of little avail if limited to their own activity, and it is only in the possibility of their almost illimitable development that their fertilising effects may be secured."

It is of interest in this connection to recall the fact that a few months ago the realisation of the prophecy above made was accomplished. There is now made, and offered for sale to farmers, a nitrifying ferment called *nitragin*, which is prepared from the tubercles of certain leguminous plants. It is found that this material is of use only when applied to crops similar to those from which it is made, while it does not act upon other crops, especially those of a non-leguminous nature. For instance, if the farmer wish to fertilise his clover-field with a nitrifying ferment prepared in this way, he must get one which is prepared from clover. If it be a field of peas or beans, on the other hand, he must secure a ferment prepared from these vegetables. This process may seem ridiculous to those who do not carefully consider all of its aspects; but in a little phial, no bigger than a goose-quill, can be

easily contained the seeds of ferments which, by proper multiplication, will produce an active nitrification over a large area. In the preparation of the ferment it is best to mix it with fine, moderately moist soil. After thorough mixing, this soil is then sowed over the land as one would sow wheat or oats. By the process of fission the organisms which are thus introduced into the soil rapidly multiply, and if they find the rootlets of plants suitable to their environment they at once attach themselves thereto, where new tubercles, similar to the ones you saw upon the screen, are formed. It is too early yet to speak of the commercial success which will attend this method of fertilisation, but there is no doubt of the fact that when a field which contains an abundance of nitrogenous matter becomes practically sterilised, this matter may be rendered more available by the introduction of proper nitrifying organisms, and it is also certain that when those crops, such as the Leguminosæ, which are suited to the development of the colonies of tubercles upon the rootlets, are seeded with the proper organisms, the number of tubercles is increased, their activity favoured, and the assimilation of atmospheric nitrogen hastened. (Photographs were exhibited upon the screen to show the influence of inoculating different plants with different ferments developed on radical tubercles).

Ferments Inimical to Agriculture.—It has been noticed by many observers that when nitric acid is subjected to certain fermentative processes it becomes decomposed and gradually disappears. In studying the causes which lead to this decomposition, it is found that it is due to the action of a micro-organism or ferment, which, by reason of the result of its functional activity, is called a denitrifying organism. While it is true that in numbers and activity this denitrifying organism does not equal its nitrifying relation, yet it is a matter of no inconsiderable importance to know fully the laws which govern its existence. As in the case of the bacteria which are found in ripening cream, where some produce evil and some good effects, so it is with those in the soil. The favouring organisms, whose functional activity prepares nitrogen in a form suited for plant food, are accompanied by others, doubtless nearly related to them, whose functional activity tends to destroy the work which the first have accomplished. It thus happens that in the fermentation of nitrogenous bodies there is danger of losing, as has already been said, a part of the nitrogen, which may either escape as gaseous oxides unsuited for the sustenance of plants, or even as free nitrogen. The object, at least the practical object, of the investigation of these denitrifying organisms, should be to discover some process by which their multiplication could be prevented and their activity diminished. At the present time all that is known is that in ordinary circumstances these organisms are not developed in sufficient numbers to prove very destructive. It has already been mentioned, however, that in case of a very great excess of organic nitrogenous matter a considerable quantity of the nitrogen therein contained may, through the action of these organisms, be lost. The practical lesson taught here is to apply nitrogenous foods in a moderate manner and avoid every unnecessary excess.

In the case of nitrifying ferments, it has been seen that nitric acid and carbon dioxide are some of the final products of bacterial activity. In the denitrifying process, on the other hand, free hydrogen and free nitrogen are the results of the final activity of the micro-organisms. In these tubes which I show you, which are partly filled with gas, the evolution of the gaseous material has been secured by introducing into the sterilised solution containing a nitrate, a denitrifying ferment obtained from a soil taken in proximity to a stable. Experience has shown that stable manures of all kinds contain these denitrifying ferments, and that these are capable of causing considerable waste of nitrogen, unless care is taken in their use. The results of such experiments as these show conclusively that it would be a useless extravagance to use a fertiliser

containing nitric acid, such as Chili saltpetre, in connection with stable manures.

Pathogenic Ferments.—There are also other forms of ferments in the soil of an objectionable nature which are not related to the nitrifying organisms. It has been observed in France that, in localities where animals are interred which have died of charbon, the germs of this infectious malady persist in the soils for many years, and that, especially when cereal crops are cultivated upon such soils, there is great danger of contaminating healthy cattle with the same disease. In one case it was observed that many sheep which were pastured in a field in which, two years before, a single animal which had died of charbon was buried, were infected with the disease and died. In like manner, it is entirely probable that the germs of hog cholera may be preserved in the soil for many years, to finally again be brought into an activity which may prove most disastrous for the owners of swine. Every effort should be made by agronomists to avoid infecting the soil by carcasses which are dead from any zymotic disease. Cremation is the only safe method of disposing of such infected carcasses. The investigations of scientists have shown that there are many diseases of an infectious nature due to these germs, and that these germs may preserve their vitality in the soil. Among others may be mentioned yellow fever and tetanus, and the microbe producing the bubonic plague, which retains its vitality in the soil, and thus escapes entire eradication.

Use of Sewage as Fertiliser.—For the reasons given above, the agronomist, who also has at heart the health and welfare of man and beast, can hardly look with favour upon any of the plans which have been proposed for the use of sewage from large cities for irrigation purposes. There is scarcely a time in any large city when some infectious disease, due to the activity of germs, does not exist, and the sewage is liable at all times to be contaminated therewith. In view of the fact that the vitality of the germs mentioned above may be continued for a long time in the soil, it is fair to conclude that it is of the utmost importance to avoid the contamination of the soil, where it is to be used for agricultural purposes, with any of the dejecta which may come from those infected with any zymotic disease whatever.

Supplying Lost Nitrogen.—It is evident that if no process of supplying the loss of nitrogen existed, the soil would soon lose its power of furnishing food and raiment for man. The philosopher who studies the system of Nature sees in the far future the advent of a time when the environment of man on the Earth will be too harsh for his present organisation. The slow cooling of the Sun, and consequently of the Earth, is the principal cause of this misfortune. But added to this must be considered the gradual disappearance of carbon dioxide and organic nitrogen, two of the essential components of the environment which makes plant-life possible. Diminishing heat and light, disappearing carbon dioxide and organic nitrogen, are, little by little, making the struggle for existence harder.

Nitrogen is lost not only by the action of the denitrifying organisms, but also by the solution of nitrates and their loss in drainage waters. From the sea this loss is restored in part by fish and sea-weeds. This is a practical illustration of the text, "Cast thy bread upon the waters, and it shall return after many days." The organisms that oxidise atmospheric nitrogen supply another part.

Fortunately, living organisms adapt themselves to changes in their environment, and life, therefore, will still be possible when the present conditions of existence shall have disappeared.

A careful study of the causes which produce a waste of nitrogen and those which restore the loss, gives the pleasing assurance that the present kind of man will not die of nitrogen hunger. Some of the best producers of proteids flourish at high latitudes.

When the last man of the present race, with a stature

diminished by long ages of hopeless labour, and with features pinched from hunger and cold, shall have been driven to the equator by the advancing armies of ice, his last look will be at the mocking disc of the Sun, denying him warmth, and his last mouthful of food will contain the proteids of oatmeal.

(The lecture was fully illustrated with experimental cultures of soil microbes and by means of lantern slides).—*Journal of the Franklin Institute*, cxliii., p. 293.

CONTRIBUTION TO THE STUDY OF HYPONITROUS ACID.*

By A. HAUTZSCH and A. L. KAUFMANN.

In taking up the study of hyponitrous acid, discovered by Maumene, and afterwards worked on by Divers, Zorn, and Van der Plaats, we had two considerations in view:—firstly, the relations existing between hyponitrous acid and the so-called dinitrogenised compounds, expressed by the formula $\text{HON}=\text{NOH}$, and by the name *dinitric acid*, proposed by Wislicenus; and secondly, the relations existing between this acid and nitramide, NH_2NO_2 , discovered by Thiele, and considered by him to be a structural isomer of hyponitrous acid, which was then unknown in the free state.

We have perfected Zorn's method of preparing this acid, in such a manner as to always ensure satisfactory results, and finally we have succeeded in obtaining free nitrous acid in the solid state. Further, we have prepared hyponitrite of ammonium, which was up till recently, hardly known, and have isolated in the solid state the benzylic ether of hyponitrous acid. The free acid, the salts, and its ether have been submitted to searching enquiry as to their chemical and physical properties, and compared as far as was possible with the *nitramide* isomer.

Formation of Hyponitrites.

In the series of the intermediate products of reduction, between nitric acid and ammonia, hyponitrous acid will be found between nitrous acid and hydroxylamine:—



It follows, then, that hyponitrous acid can be obtained either by the reduction of the preceding or the oxidation of the following compound in the series.

a. *Methods of Reduction.*—Sodium amalgam, either directly (Divers, *CHEMICAL NEWS*, xxiii., p. 206; Zorn, *Berichte d. Ch. Ges.*, x., p. 1306) or indirectly, has been principally used as the reducing agent; that is to say, by submitting to electrolysis aqueous solutions of nitrite of soda, with the mercury as the negative pole (Zorn, *Berichte*, xii., p. 1509).

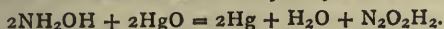
However, the resulting amounts indicated by Zorn have never been obtained by the different chemists who have studied this reaction; but Tanatar (*Berichte*, xxvi., p. 163) succeeded in obtaining twice as much as Divers by using a liquid sodium amalgam.

The assertions of some writers that it would be possible to obtain hyponitrite of soda by treating nitrite of soda with ferrous hydrate (Zorn, *Ibid.*, xv., pp. 1007 and 1288) or by melting nitrite of soda with scrap sheet-iron (Mencke, *CHEM. NEWS*, xlix., p. 45) have not been confirmed. Similarly, we have not been able to prepare hyponitrites by the reduction of nitrites in an aqueous solution, by means of aluminium amalgam, as described by H. Wislicenus and L. Kaufmann (*Berichte*, xxviii., p. 1323).

b. *Methods of Oxidation.*—Hyponitrous acid can be prepared by the oxidation of hydroxylamine and its derivatives. The formation of hyponitrous acid, observed by Thum, by the action of hydroxylamine on some metallic oxides,

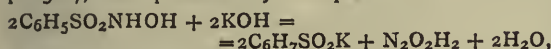
* *Moniteur Scientifique*, vol. xi., p. 336, May, 1897.

—mercuric oxide, cupric oxide, and oxide of silver,—is based on the direct oxidation of hydroxylamine:—



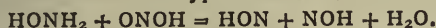
The quantities obtained by this method are not so insignificant as stated by Thum. It was by thus treating, with an excess of mercuric oxide, an alkaline solution of hydroxylamine, prepared from 5 grms. of sulphate of hydroxylamine and an excess of caustic potash, that we obtained—after eliminating the mercury, neutralising with nitric acid, and precipitating with nitrite of silver—0.5 gm. of hyponitrite of silver. This quantity represents 10 per cent of the weight of the sulphate of hydroxylamine, and about 5 per cent of the theoretical returns.

The splitting up of benzo-sulphydroxamic acid (obtained by the action of hydroxylamine on sulpho-chlorated benzene) by alkalis, into benzo-sulphuric acid and hyponitrous acid, lately observed by Piloty (*Berichte*, xxix., p. 1560), and represented by the equation—



can also be considered as an oxidation of hydroxylamine. This method of preparing hyponitrous acid gives fairly satisfactory results.

The action of nitrous acid on hydroxylamine also gives rise to the formation of hyponitrous acid,—



According to Wislicenus (*Ibid.*, xxvi., p. 771) this reaction might be considered as a condensation. Neither the action of nitrite of soda on sulphate of hydroxylamine (Wislicenus) or that of nitrite of silver on chlorhydrate of hydroxylamine (Kratschmer, *Thèse*, 1895, p. 9) give satisfactory results. The method proposed by Tanatar (*Berichte*, xxvi., p. 763)—reaction in the presence of certain bases—seems to be the one giving the best results.

We have endeavoured to make hyponitrite of soda, and potash, by passing a current of dry nitric oxide through a liquid sodic or potassic amalgam. But the result was nil; there was no reaction. Equally unfruitful were the attempts of P. Schatzmann to combine protoxide of nitrogen with caustic potash under pressure, or at a high temperature. The only products of this experiment were ammonia and nitrite of potash, but no hyponitrite of potash.

Preparation of Hyponitrite of Silver.

As in our experiments we had need of large quantities of hyponitrite of silver, which serves as the starting-point for the preparation of free hyponitrous acid, we endeavoured to improve the then best known among the methods of preparing hyponitrite of silver, viz., that based on the reduction of nitrite of soda by sodium amalgam. We have found from experience that the return is considerably increased—1. When working at a low temperature; 2. When the nitrite of silver is present in great excess with regard to the nascent hydrogen; 3. When the reaction is carried on in alkaline solution, hyponitrous acid being unstable except in strongly alkaline solutions. Influenced by these facts, we propose the following method for the preparation of hyponitrite of silver:—

To a properly cooled solution of 20 grms. of pure nitrite of soda add 10 grms. of caustic soda in 200 c.c. of water; a liquid amalgam, obtained by dissolving 16 grms. of sodium in 2800 grms. of mercury is slowly added, drop by drop. During the operation, which should last about three-quarters of an hour, the mixture should be strongly and continually agitated. After having separated the mercury, the liquid, cooled to 0°, is heated with diluted nitric acid until it gives but a very slight alkaline reaction. To eliminate the hydroxylamine, which is always formed in this reduction, mercuric oxide is added, the mixture being still constantly stirred, until the last portion of oxide added no longer changes colour. The liquid is rapidly filtered with a filter-pump, a small quantity of mercuric

oxide being first placed in the filter. The filtrate is then neutralised with nitric acid, and precipitated with nitrate of silver. The yellow precipitate obtained is washed with warm water, first on the filter, then by decantation in an Erlenmeyer flask, re-dissolved in very dilute nitric acid, and cooled down to 0°. The solution is then rapidly filtered and precipitated by ammonia; by repeating this we obtain perfectly pure hyponitrite of silver. This can be dried either over a water-bath or in an exsiccator after being washed with alcohol and ether.

Analyses of the substance thus obtained have given the following results:—

		I. Grms.	II. Grms.
Substance used		0.2019	0.3308
Ag by titration		0.1573	0.2580

	Found.	Theory.
	I. II.	
Ag ..	77.91% 78.00%	AgNO .. 78.26%

The salt prepared as described above possesses all the properties of hyponitrite of silver. But, contrary to the statement of Van der Plaats, it does not detonate at 150°. In spite of all our efforts we have been unable to explode it. It is also worthy of note that this silver salt keeps its yellow colour in diffused light; but in direct sunlight it quickly assumes a yellowish green colour, and when moistened the surface becomes black.

The amount of hyponitrite of silver obtained, starting with 20 grms. of nitrite of soda, is from 2 to 3 grms., or 6 to 7 per cent of the theoretical quantity. These figures correspond to the maximum returns indicated by Zorn, but they have not been obtained by any other experimenters who have used Zorn's method.

It is best not to attempt the reduction on more than 20 grms. of nitrite of silver at a time. But the rough products resulting from several reductions can all be purified together. In one or two days one can easily prepare 20 grms. of pure hyponitrite of silver. We therefore believe that the method we have just described is the simplest and the least expensive.

(To be continued).

SOME EXPERIMENTS WITH CATHODE RAYS.*

By A. C. C. SWINTON.

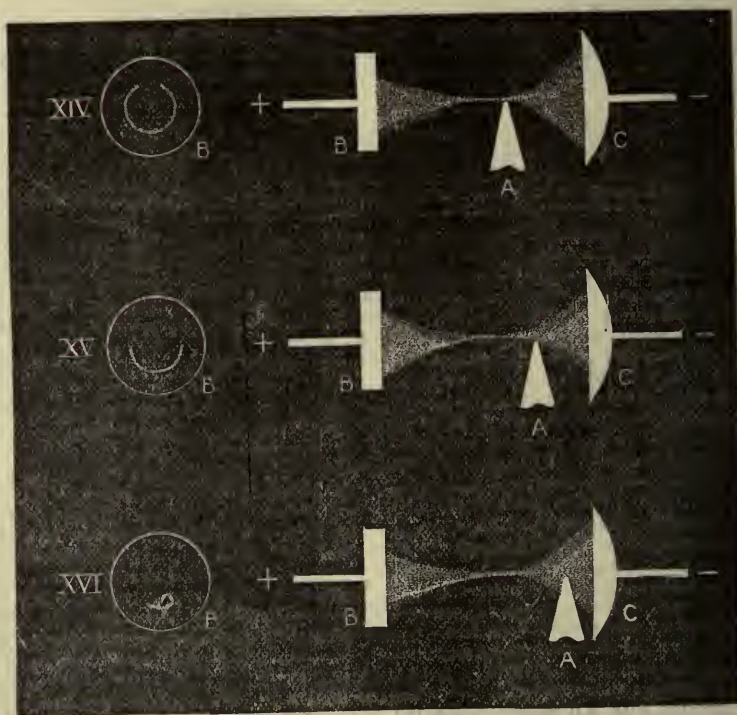
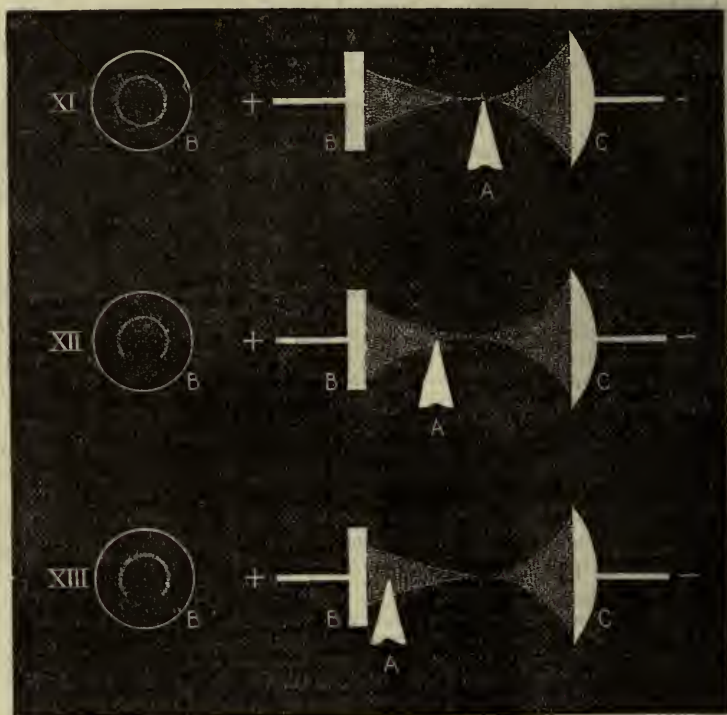
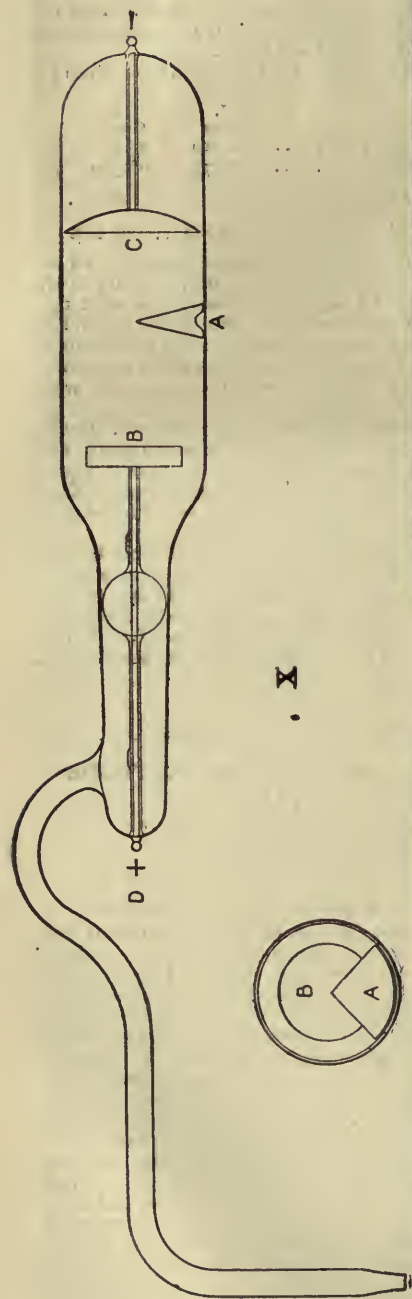
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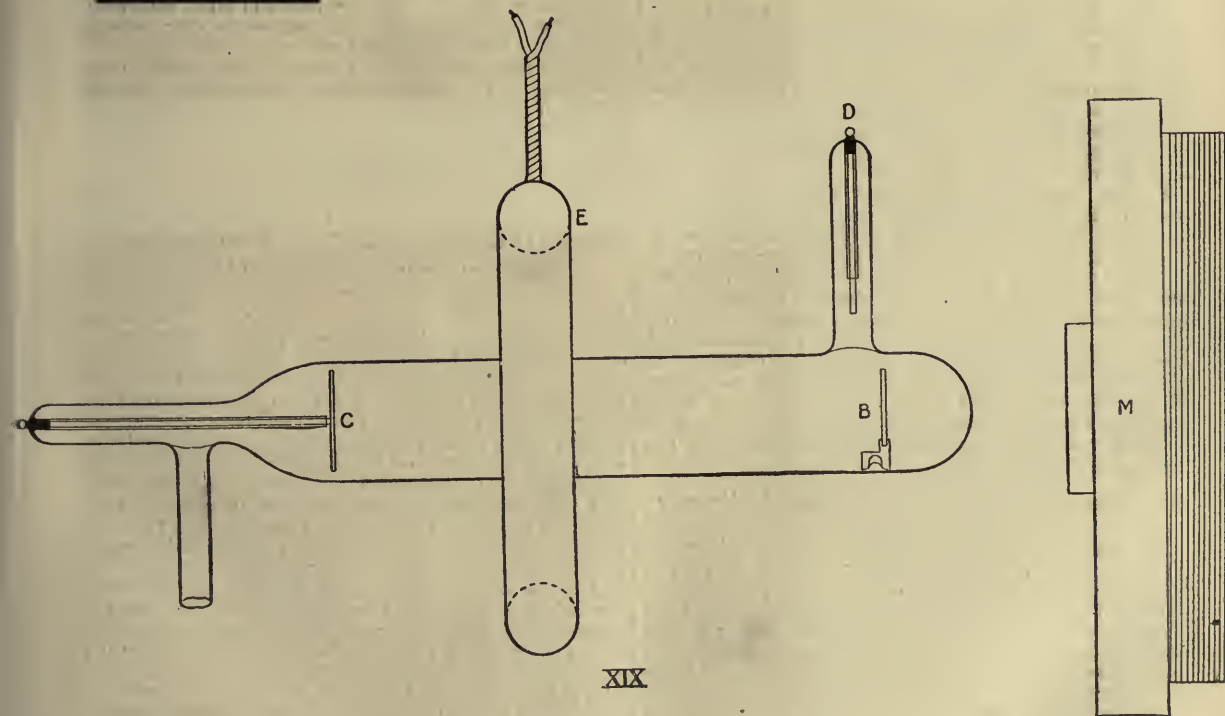
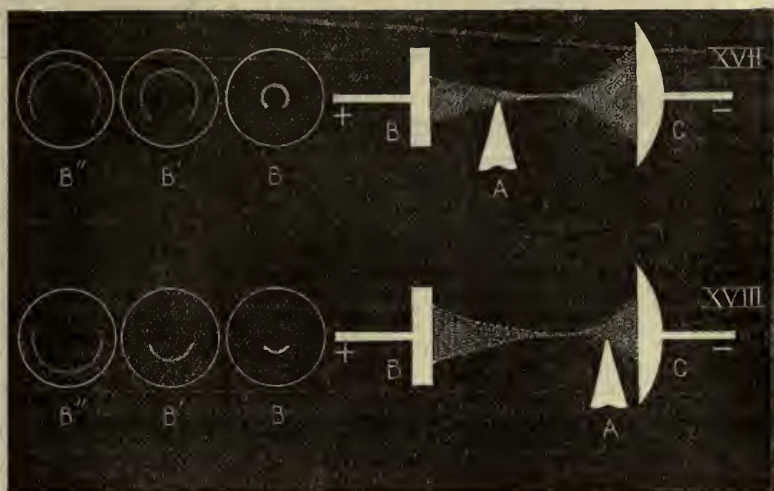
The Rays cross at the Focus with no Rotation.

In order to investigate the cathode rays in a focus tube still further, and more especially in order to discover whether the various rays from the cathode cross one another at the focus, or diverge again without crossing, and also in order to discover whether there is any twist or rotation of the rays, similar to what has been observed in the case of rays focussed by magnetism,† a tube was constructed similar to that used in the previous experiments, with a carbon anticathode which was also the anode, fixed at the opposite side of the focus from the cathode, with the focus about equally distant between it and the cathode. The peculiarity of this tube consisted in the fact that a sector of the aluminium cathode, equal to one-eighth of the total area of the cathode, had been entirely removed, as shown at c, fig. 9. It was expected that on using this tube, with the proper degree of vacuum to form a well-defined ring on the anti-cathode screen, that a portion of the ring, corresponding with the amount of the cathode cut away, would be found wanting; and that by the position of this gap in the ring it would be possible to ascertain whether the rays crossed at the focus, and whether there was any rotation. What

* A Paper read before the Royal Society, March 11, 1897.

† See experiments by K. Birkeland, *Electrical Review*, June 12, 1896.





actually was observed is shown for three different conditions of vacuum in fig. 9, B being for the highest, and B'' for the lowest vacuum. As will be seen, the expected gap in the ring was obtained, but with the unexpected addition that the dimensions of this gap, instead of being only one-eighth of the circumference of the ring, was seven-eighths of the circumference. In fact, the amount of ring shown corresponded not with the seven-eighths of the remaining cathode surface, but with the one-eighth of the cathode that had been removed. The portion of

ring that did appear was of a length corresponding exactly to the arc of the removed sector of the cathode, according to its greater or lesser nearness to the centre with different conditions of vacuum; and as the portion of ring was in each case exactly in line with the portion of cathode that had been cut away, it would appear that there is no rotation of the cathode beam as a whole, that the rays do cross at the focus; and, further, that when the hollow convergent cone is, as it were, split in this manner, some unexplained action, similar in effect to the

existence of a circular surface tension, causes the gap to widen out and the remaining portion of the ring-shaped section of the cone to contract correspondingly, without, however, altering its diameter.

In order to further investigate the matter another tube was made, as shown in fig. 10, in which the concave cathode was complete; but the interior of the tube was furnished with a small movable piece of aluminium, A, which by shaking could be moved up and down the tube between the cathode, C, and anti-cathode, B, and which, while not quite reaching the centre of the tube, would fill up very nearly one-quarter of the circular sectional area of the latter.

With this arrangement of tube, with the aluminium obstacle placed just at the focus, as shown in fig. 11, the point of the obstacle just missing the cathode rays, a complete ring was formed on the carbon anti-cathode. On moving the obstacle slightly into the divergent cone, exactly one-quarter of the ring on the anti-cathode failed to appear, as shown in fig. 12, and on the obstacle being further moved in the same direction the result was not altered, as shown in fig. 13.

As in each of the latter two cases there was no displacement of the gap in the ring, the above showed that there is no rotation of the divergent cathode cone.

Experiments were next tried with the aluminium obstacle, moved so that its point just entered the convergent cone of cathode rays, when a small portion of the ring was cut out; but on the opposite side, as shown in fig. 14, this confirming the previous experiments, which showed that the rays cross one another's paths at the focus without rotation. Upon moving the aluminium obstacle a little nearer to the cathode, so that its point entered still further into the convergent cathode beam, one-half of the ring disappeared, as in fig. 15, while when the obstacle—which, it should be remembered, blocked only one-quarter of the circular area of the tube—was brought close up to the cathode, only about one-quarter of the ring remained, as in fig. 16.

Further experiments were tried with the aluminium obstacle both in the divergent and convergent cones, but with the tube exhausted to different degrees of vacuum, the result being as shown in figs. 17 and 18, in which in each case B shows the highest vacuum and A the lowest, from which it will be observed that when the obstacle was in the divergent cone a portion of the ring was cut off exactly proportional to the angle subtended by the sides of the obstacle; while when the obstacle was placed in the convergent cone, a much larger proportion of the ring was cut off in each case, this being much more marked with a high vacuum when the diameter of the ring was small than with a low vacuum when the diameter of the ring was large.

Convergent and Divergent Cones produced by Magnetic Focussing.

In order to discover whether the apparent hollowness of the convergent and divergent cones of cathode rays as above observed, when the focussing was performed by means of a spherical cathode, was in any way due to the concave form of the cathode or to the fact that the rays were converging or diverging, experiments were tried with a tube having a flat aluminium cathode, the rays being caused to converge to a focus by means of a powerful electro-magnet in the manner described by the writer in his paper on "The Effects of a Strong Magnetic Field upon Electric Discharges in *vacuo*" (*Roy. Soc. Proc.*, 1896, vol. lx., p. 179).

The arrangement is shown in fig. 19, the carbon anti-cathode screen, B, being movable, and not connected to the anode D, which was contained in an annex to the tube. By increasing or decreasing the power of an electro-magnet, M, by moving it nearer to or further away from the tube, and by moving the anti-cathode screen up and down the tube, the cathode rays could be focussed on the anti-cathode screen so as to form a circle of any desired

size, the focus, which appears to be exactly on the pole of the magnet, being, of course, always beyond the anti-cathode.

In order similarly to investigate a divergent cone of cathode rays magnetically produced, a circular coil of wire, E, was employed instead of the magnet in the manner recently described by Professor Fleming (*Electrician*, January 1, 1897). This coil, which had 72 turns of No. 18 S.W.G. size wire, was supplied with 20 to 25 amperes of current from a storage battery. It focussed the cathode rays at a point exactly central to its own plane, from which they again diverged on to the anti-cathode screen.

With convergent and divergent cones of rays produced magnetically in the above manner, there was no difficulty in showing that, under suitable conditions, these cones acted as if they were hollow, giving bright rings of varying sizes, sometimes with and sometimes without bright central spots, upon the carbon anti-cathode screen exactly similar in appearance to those obtained with the concave cathode.

Further observations were as follows:—

In some instances, two concentric hollow rings were observed, especially with a low vacuum when the magnet was suddenly turned on or off. The rings are probably not simultaneous, but successive, but this cannot be detected with the unaided eye.

With a high vacuum, and the magnet so arranged as to focus the rays accurately upon the carbon, a small bright spot appears at first; as the vacuum goes down this point becomes larger and fainter, but still solid. Suddenly it becomes hollow and brighter, then, as the vacuum falls still further, the ring becomes solid again, though larger and more faint than before, finally it disappears. After this stage it can be reproduced momentarily, without alteration to the vacuum, by switching the magnet on and off suddenly, when it is usually hollow, but sometimes solid.

(To be continued).

SEPARATION OF CHLORINE AND BROMINE.

By H. BAUBIGNY and P. RIVALS.

DETERMINATIONS of chlorine, bromine, and iodine in mixtures of haloid salts have been always considered as operations of great difficulty. They were, in fact, possible only by indirect methods, generally inaccurate and not admitting of any control. If we can now separate with accuracy iodine from the two other elements, the procedures given, even in the last few years, for determining chlorine in presence of bromine, or inversely, are still imperfect. This fact has led us to our present researches. We shall consider at first the case of a mixture of chlorides and bromides, where the problems are to bring the chlorine and the bromine to the state of alkaline salts, or, secondly, to the state of silver salts.

Alkaline Salts.—The modern methods of separating the halogens are founded in general upon the different properties of their hydric acids in presence of different oxidising agents, the effects of which differ also with the conditions of the experiment. As oxidisers we have employed successively lead and manganese peroxides, oxygenated water, the chromates, permanganate, ammonium persulphate, the arseniates, &c., most frequently in presence of a small quantity of sulphuric or acetic acid, or of a salt easily decomposed, such as ferric or aluminic sulphate. But in any case we must meet the necessity of having a solution so dilute that the oxidation may be limited to one of the hydric acids. Of all these oxidising agents, those which are the most soluble are evidently the most perfect, or at least the most regular, in their action; and of these, permanganate is one of the most energetic. Peon de St. Gilles has shown that if we treat a mixture of alkaline haloid salts in a neutral or alkaline

liquid with an excess of permanganate, all the iodine—and the iodine alone—is oxidised, so that the iodide is entirely converted into iodate, whilst the alkaline chlorides and bromides are not affected even at 100°. But if the permanganate does not act upon potassium or sodium bromides, nothing proves that it must be the same for those of all metals. Indeed, we have found that if the solution of neutral copper chloride is not attacked by permanganate in the cold, that of the bromide of the same metal is decomposed with liberation of bromine.

The phenomenon of oxidation is not of the same order as for the alkaline iodides, but it permits us to separate bromine and chlorine; for in an alkaline chloride or bromide the addition of neutral copper sulphate, in virtue of the law of distribution, determines the formation of copper chloride or bromide. If, then, we further add a little permanganate, the bromine, if present, is displaced. In these latter conditions it is easy to demonstrate that the reaction is quantitative.

To this end, having prepared standard solutions of alkaline chloride and bromide, we operated with these liquors, at first separately, and then in mixing them in known proportions. In our experiments the quantities of salts varied from 0.120 to 0.400 grm., and for these weights we have taken from 4 to 8 grms. copper sulphate, and from 0.350 to 0.400 grm. permanganate. We commence by dissolving the sulphate, $\text{CuSO}_4 + 5\text{H}_2\text{O}$, with the alkaline salt, and in the cold liquid the crystals of permanganate. The vessel is then set in a vacuum at the ordinary temperature (15–18°) over fragments of potassa. The next day the dry residue, if taken up in a little water, gives off no odour. We then complete the solution by adding enough sulphurous acid to reduce MnO_2 and what remains of the permanganate, and in the liquid we precipitate the chlorine or bromine by silver nitrate, very strong in nitric acid to prevent the reduction of a little nitrate by the excess of sulphurous acid, which is destroyed if heated in presence of nitric acid.

We collect the bromide or chloride until the liquid is cold—for silver chloride is especially soluble in boiling water, even if neutral.

The following are some of the results obtained:—

Value in silver salt of KBr used.	AgBr recovered.
0.192 grm.	0.0005 grm.
0.384 „	0.0006 „
Value in silver salt of the NaCl used.	AgCl recovered.
0.2922 grm.	0.586 grm.
0.2917 „	0.5858 „

On the subject of experiments with chloride, we must add that for each of them the dry residue has been re-moistened and re-dried twice *in vacuo*, so as to exaggerate the decomposition of the chlorides if such were capable of being produced; the action may therefore be regarded as nul.

Lastly, for mixtures of chlorides and bromides experiment showed—

Value in silver salt—		Salt of silver recovered.
Per NaCl used. Grm.	Per KBr used. Grm.	
0.5844	0.192	0.5854
0.0731	0.576	0.0730

—Comptes Rendus, cxxiv., No. 16.

Some New γ -Cetonic Acids.—T. Klobb.—The alcoyl-phenacylcyanacetic ethers, are easily saponified in the cold by alkalis, but if warmed they quickly give off ammonia. By this means nitrogen-free acids are obtained, of which one only—ethylphenacetylacetic acid—had up to now been prepared. Several others have been now made, and their properties are described in this paper.—Bull. de la Soc. Chim. de Paris.

NOTICES OF BOOKS.

The Electric Furnace. ("Le Four Électrique"). By HENRI MOISSAN, Membre de l'Institut. Paris: G. Steinheil. 1897. Pp. 385.

(SECOND NOTICE).

THE first application of the electric furnace, after it had passed from the experimental stage, was to the study of the crystallisation of the metallic oxides, then the volatilisation of several simple bodies. For the first of the above-named experiments a furnace of quicklime was used, and when working on small quantities of material, and only using about four horse-power, the carbons were purified by being first submitted to the action of chlorine at a high temperature, and then cooled in a current of nitrogen. But when using such currents as were furnished by thirty to thirty-five horse-power, the electrodes had to be prepared and purified with the minutest care, small amounts of impurities exercising great influence on the results. A great variety of substances were experimented on, such as chalk, lime, strontia, baryta, magnesia, alumina, &c. It is impossible to operate on a small quantity of alumina in contact with lime, as a liquid aluminate of lime is immediately formed; in this case a carbon crucible is used. By adding a very small quantity of sesquioxide of chromium to the alumina, a mass of small crystals of veritable rubies is formed. These, however, are not so fine as those prepared by MM. Frémy and Verneuil. The author has not followed up this matter.

The oxides of the iron group stable at high temperatures form masses bristling with small crystals.

M. Dufau has, by using the electric furnace, noted the existence of a chromite of lime, $\text{Cr}_2\text{O}_3 \cdot \text{CaO}$; a tetra-chromite of barium, $4\text{Cr}_2\text{O}_3 \cdot \text{BaO}$; a cobaltite of magnesium, $\text{CoO} \cdot \text{Mg}$; and a nickelite of barium, $2\text{NiO}_2 \cdot \text{BaO}$.

The ordinary metals are easily volatilised and condensed, as well as even such refractory ones as platinum and uranium.

The experiments to try and volatilise boron were not successful, as boride of carbon was immediately formed; this is easily melted, and on cooling forms definite crystals.

An early conclusion arrived at by the author is, that at such high temperatures as those obtained by means of his furnace, the bodies hitherto considered as most refractory are easily volatilised, and those looked upon as being the most stable in mineral chemistry are destroyed either by dissociation or volatilisation. There remains only a series of new compounds, perfectly crystallised and of exceptional stability, which are able to resist the action of this extreme amount of heat. These are borides, silicides, and—above all—the metallic carbides.

Carbon is, of all the simple bodies, the one which forms the most curious allotropic varieties. Its contradictory properties, its different specific heats, &c., have long been matters for earnest thought and study. The study of amorphous carbon ranges over a wide field, and we will do no more than mention the fact *en passant*. Graphite, before the researches of M. Berthelot, included all varieties of carbon capable of leaving a mark when rubbed on paper; molybdenite was thus easily confounded with graphite. Berthelot defined graphite as "every variety of carbon capable of forming by oxidation a graphitic oxide." This property definitely established the classification of the varieties of carbon into three groups—diamond, graphite, and amorphous carbon. M. Moissan has, in his researches, shown the possibility of bringing all kinds of carbon, diamond or amorphous, into the graphitic state. The discovery of graphite in a meteorite from the Cañon Diablo led the author to examine other meteorites, to find out if they also contained carbon, and, if so, in what form. In some, such as the meteoric iron from Kendal County, Texas, none was found; that from Newstead, in Roxburghshire, contained

amorphous carbon and graphite; déesite, from Chili, contained a small quantity of graphite; and of three samples of iron from Ovifak, all contained amorphous carbon, two contained graphite, but none contained diamond, either black or transparent.

A few of the graphites which can be prepared in the laboratory are here passed in review. Diamond heated in the electric arc is converted into graphite; wood charcoal, properly purified, is, at a temperature of about 2200°, converted into graphite in ten minutes. There are many other methods for producing it at densities varying from 2·10 to 2·25.

The study of the solubility of carbon in metals at high temperatures led to the research on the action of boron and silicon on liquid carbide of iron, and it is shown that these chemical reactions are as clear and well-defined as those in aqueous solutions, as performed in the laboratory at ordinary temperatures. A sample of cast-iron was taken, containing 3·18 per cent of carbon and 0·5 per cent of slag. To 10 grms. of this was added 2·3 grms. of boron. After heating, it was allowed to cool, when the slag on the surface was found to contain all the original slag and most of the boron, while the carbon in the iron was reduced to 0·27 per cent, the boron present amounting to 8 or 9 per cent.

Silicon has a similar action of replacing a part of the carbon, the carbon expelled being found on cooling, on the top of the metal in the form of graphite.

To obtain pressure in the manufacture of graphite in molten iron, recourse was had to the plan of suddenly cooling the whole mass in cold water; this has the effect of strongly compressing the carbon dissolved in the iron, and after cooling, dissolving the iron, &c., crystals of graphite, of a beautiful shining black colour, were formed, bearing a close resemblance in general appearance to specimens of graphite found in the blue ground from Kimberley; its density is 2·16, and it burns in oxygen at about 660°.

All the samples of graphite hitherto examined have been found to contain hydrogen. This might be due to three reasons: it may be a physical phenomenon, or condensation of hydrogen gas in the graphite—as might be imagined from some experiments made by M. Cailletet, who noticed that hydrogen was absorbed by molten iron; or, again, it might be a chemical phenomenon; an experiment here described, however, shows that hydrogen is not in combination with the graphite.

Having succeeded in making crystallised graphite, it was but natural that M. Moissan should next attempt the artificial production of diamonds. Many workers have already attempted this difficult task, but their results have been contradictory and uncertain. The author fully recognised that, even if he did succeed, the diamonds obtained would, at first at any rate, be merely microscopic.

It is interesting here to note the definition of "diamond"; it is an elementary body, of maximum hardness, 3·5 density, and burns in oxygen at a temperature somewhat above 700°; 1 grm. giving 3·666 grms. of carbonic acid. The principal characteristic of the diamond is its great hardness. Numbers of bodies—carbides, borides, silicides, &c.—have been produced by means of the electric furnace which will scratch ruby, but only boride of carbon will even slowly scratch a diamond.

By studying the occurrence of diamond, and the character of its associates—zircon, topaz, titanite iron, &c.,—which are found in the same matrix and under similar conditions, M. Moissan concluded that the diamond was formed in the interior of the earth under conditions of great heat and enormous pressure. To imitate this he had recourse, as before, to the sudden cooling of a mass of molten iron in which carbon was held in solution; the exterior being suddenly solidified subjected the still liquid interior to very great pressure.

It was not without a certain amount of apprehension that this experiment was performed for the first time; indeed, it seems marvellous that the act of plunging a

crucible containing molten iron heated to a temperature of over 3000° into a vessel of cold water did not result in a disastrous explosion; but happily it is not so—the experiment has been performed over 300 times without accident.

CORRESPONDENCE.

ACTION OF ENZYMES UPON STARCHES.

To the Editor of the Chemical News.

SIR,—I have read with much pleasure the interesting review in your issue of April 30th on Dr. Stone's report, especially in regard to the action of enzymes on starches. I notice that Taka-diastase has claimed the attention of Dr. Stone to such an extent that, although this special digestive agent had not been long before his notice when he made his report, he considered it to be of sufficient importance to call for special mention and space. I am not surprised at this keen interest in regard to a preparation possessing diastatic potency to the extent present in Taka-diastase.

As I have experimented with this diastatic ferment and considered its action to a greater extent than has perhaps any one else at present in this country, I venture to place before your readers a few remarks which suggest themselves to me after making numerous tests on different varieties of starch and comparisons with other preparations for which diastatic power is claimed, as also after having had frequent discussions with medical men who have used Taka-diastase practically in amylose dyspepsia—a form of dyspepsia which is said to constitute about three-fourths of the indigestion due to deficiency in the quality or quantity of the digestive secretions generally.

When making demonstrations in this connection before some members of the British Medical Association in the summer of 1895, I was impressed by the interest which was manifested in the treatment of mal-digestion or want of due assimilation of starchy foods. This interest is of comparatively recent growth, as is much of the knowledge in regard to the causes which lead to the production of the condition referred to; in fact, not many years ago there was hardly any serious attempt at differentiation in the diagnosis or treatment of what is called "indigestion." In the minds of many persons who have studied the questions connected with this subject there still exist certain misconceptions which prevent a clear understanding of the difficulties that present themselves. Dr. R. G. Eccles states that "one of the gravest mistakes we have made in the past has been in supposing that the stomach only had to deal with proteids—that within it only albumen, casein, gluten, and the like were disposed of. The fact is that the very first important act performed in the stomach is the digesting of starch. All gluten reaches it enveloped in an insoluble coating like the sugar or gelatin on the outside of a pill. To make way for the digestion of proteids this must be rendered soluble and removed. To do so, starch-digesting has to be the first task of the stomach; following it comes proteid digestion." He refers to the belief once entertained almost universally that conversion of the trifling amount of starch acted on during the process of mastication was all the duty saliva performed, and then states that "Ptyalin was never intended by Nature to do its work in the mouth." The fact is that, under normal conditions, the contents of the stomach may not contain any free hydrochloric acid until after the expiration of half-an-hour to an hour from the time the food is ingested.

The claim made for Taka-diastase is that it will convert one hundred times its own weight of starch into assimilable material in ten minutes, and, as it is an enzyme or unorganised ferment (the action of which might theoretically be regarded as infinite), it might be considered to

proceed indefinitely if the products of its conversion were removed as formed. But Sir William Roberts states that diastase is no exception to the rule in physics, that energy in performing work is expended and finally exhausted. Nevertheless Taka-diastase converts over 1500 times its own weight of starch in three hours.

It may be argued that as digestion of starch is not likely to proceed for three hours in the stomach it is not necessary, in considering digestion, to estimate the power of a diastase during this period of time; but we have to bear in mind that, as digestion proceeds, the pylorus relaxes, allowing the passage to the intestine of finely divided particles and of such fluids as may not have been absorbed in the stomach; and as the diastase passing with such material in the early stage of digestion may still be active (as a matter of fact Taka-diastase converts starch even in a slightly acid medium), we have no reason to conclude that the action does not, under such circumstances, proceed in the intestines, where any acid may be expected to be gradually neutralised before the alkaline condition asserts itself.

About two years ago I witnessed many experiments performed by Mr. Takamine (assisted by Mr. E. V. Hitch, of Chicago) in regard to the action of Taka-diastase on different varieties of starch, as also in comparison with other diastases. The tests were almost entirely confined to the action of the diastase from the commencement of the process to the end of from twenty to thirty minutes only (that being considered a satisfactory period in which to make the tests), during which time Taka-diastase was found to be considerably more active than any of the numerous preparations with which comparison was made and for which diastatic potency was claimed. Here I would remark in regard to tests of diastase *in vitro* that there is no reason to consider that conversion to the achromic point, although useful as a means of comparison, must occur before absorption takes place in normal digestion. Sir William Roberts states that "the dextrines, even those coloured by iodine, are highly diffusible," and that complete conversion would require that the material should retrace its steps to some extent in being converted into glycogen.—I am, &c.,

THOMAS CHRISTIE.

Chepstow Place, W., May 10, 1897.

BENDING ALUMINIUM TUBES.

To the Editor of the Chemical News.

SIR,—In the paper by Mr. T. H. Norton on "The Use of Aluminum for Condensers" (CHEMICAL NEWS, vol. lxxv., p. 221) it is suggested that to bend the aluminium tube it should be filled with lead, which is afterwards melted out. Would not resin or shellac be more easily manipulated, while the traces left in the tube could be readily removed by alcohol or some other solvent? The cycle-makers, in bending steel tubes up to 1 in. diameter, habitually use resin for filling the tube previous to bending.

I should like to ask why American writers usually speak of "aluminum." The name "aluminium" has had a long and honourable career, and it seems a pity, as Curran would have said, to knock out one of its *i's*. The oxide is, and has always been, called alumina; but no one, I believe, has proposed the names "sodium" and "potassium" because *their* oxides are called soda and potassa.—I am, &c.,

E. G. BRYANT.

King's School, Pontefract, May 8, 1897.

Death of Mr. M. Carey Lea.—We regret to hear of the death of Mr. Carey Lea, at Philadelphia. The deceased was well known to the readers of the CHEMICAL NEWS in connection with his researches on the allotropic forms of silver and the photo-sensitive compounds of this metal.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société Chimique de Paris.

Series 3, vols. xvii. and xviii., No. 7.

Apparatus for the Estimation of Free Nitrogen in Purified Coal-gas.—G. Arth.—The author has arranged his apparatus in such a manner as to enable him to collect and measure large quantities of nitrogen gas, thereby considerably reducing the effect of *errors of observation*. The apparatus, which appears to give excellent results, may be briefly described as a kind of combination of Crum's nitrometer with Frankland's gas measuring apparatus, but cannot be properly understood without the accompanying diagram.

New Fractionation Apparatus for Use in Industrial Laboratories.—A. Tixier.—This apparatus is an improvement on previous forms, but requires the accompanying diagram.

Cryoscopic Measurements.—A. Ponsot.—The author has made a number of experiments to determine the congelation-point of solutions of cane-sugar of various strengths. The thermometer used was an extremely delicate one, each principal division being equal to 0.5 m.m.; but by very fine subdivision, and using a glass magnifying fifty times, and allowing corrections for pressure and calibration, the final readings, he claims, can be reduced to 1/20,000th of a degree Centigrade.

Ethylisoamylamines.—Aug. Durand.—These were obtained by the action of iodide of ethyl on isoamylamine. By mixing them carefully in certain proportions a pasty mass is formed, composed of the iodides of the primary, secondary, and tertiary bases. This mass is dissolved in water, boiled, and a solution of potash added, then boiled again; the distillate is received in an excess of dilute hydrochloric acid. From this, the secondary base—that is to say, ethylisoamylamine—can be separated in the form of its nitroso-derivative by adding nitrite of soda dissolved in a small quantity of water. This is, after purification, treated in various manners. Several of its compounds are here described, such as chloroplatinate of ethylisoamylamine, chloroaurate of ethylisoamylamine, oxalate of ethylisoamylamine, &c.

Anethol and Homologues of Anethol.—Ch. Moureu and A. Chauvet.—The authors have succeeded, with great ease, in preparing two homologues of anethol, viz., parabutenylanisol and paraisopentenylanisol.

Yellow Colouring-matter derived from Dinitrofluoresceine.—F. Reverdin.—A paste is made of 100 grms. of water and 100 grms. of dinitrofluoresceine; to this is added, while agitating, 75 c.c. of 21 per cent ammonia. The mixture dissolves at once with an increase of temperature; it then gradually commences to thicken, till after a few hours it forms a solid mass. It is then triturated with 125 c.c. of salt water, filtered; then, after well draining, the product—an ammoniacal salt of the colouring material—is converted into an acid by weak hydrochloric acid. It is filtered, dried, and then digested for some hours at the ordinary temperature with 10 parts of acetone, in which it is completely insoluble, but which frees it from several impurities and by-products which accompany it. After filtering and drying it is converted into a salt of soda easily soluble in water. About 70 grms. of this salt are obtained. The first filtrate contains an orange colouring-matter, which no longer gives the reactions of dinitrofluoresceine, but which, by treating with bromine, gives a red colouring-matter, similar to scarlet.

Some Derivatives of Furfurane.—P. Freundler.—Several derivatives of furfurane have not yet been

described, in spite of the interest there would be in comparing them with the corresponding benzenic derivatives. Among these are *furfurane-amine* or *furane-amine*. The best method of preparing this body was found to be by the nitration of furfuran and the reduction of the nitrified derivative. This will be fully described in a future paper, the present one being devoted to the action of the hypobromites and of hydrazin on the pyromucic derivatives.

Distillation of very Dilute Mixtures of Ethylic Alcohol and Water. Application to the Estimation of Alcoholic Solutions containing only 1/3000th to 1/10,000th part.—M. Nicloux and L. Baudeur.—This paper will be inserted in full.

Revue Universelle des Mines et de la Metallurgie.

Series 3, Vol. xxxviii., No. 1.

This issue contains no chemical matter.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Wells' Patent Continuous Cooling Process.—Will some correspondent kindly inform me who are the proprietors of this process.—ENQUIRER.

MEETINGS FOR THE WEEK.

MONDAY, 17th.—Society of Arts, 8. (Cantor Lectures). "Design in Lettering," by Lewis Foreman Day.

WEDNESDAY, 19th.—Society of Arts, 8. "London Water Supply," by Dr. P. F. Frankland, F.R.S.

THURSDAY, 20th.—Chemical, 8. "Theory of Osmotic Pressure and the Hypothesis of Electrolytic Disassociation;" "Molecular Rotation of Optically Active Salts;" "Heats of Neutralisation of Acids and Bases in Dilute Aqueous Solution;" by Holland Crompton, "The Platinum-Silver Alloys—their Solubility in Nitric Acid," by John Spiller. "A Comparative Crystallographical Study of the Normal Selenates of Potassium, Rubidium, and Cæsium," by A. E. Tutton.

Society of Arts, 4.30. "Kerman and Persian Beluchistan, with special reference to the Journeys of Alexander the Great and Marco Polo," by Capt. P. Molesworth Sykes.

WANTED.

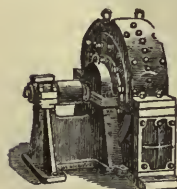
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VOL. LXXV., No. 1956.

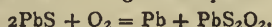
ON THE REACTIONS BETWEEN LEAD AND THE OXIDES OF SULPHUR.*

By HENRY C. JENKINS and ERNEST A. SMITH,
Royal College of Science, London.

THE authors of this paper draw attention to the fact, previously noticed by one of them, that when molten lead is exposed at high temperatures to the action of a current of sulphur dioxide, both lead sulphide and lead oxide are found in the product.

The paper is the account of a research undertaken with a view to ascertain the precise reaction which takes place under these conditions, as well as to see whether it did not account for some anomalies that are met with whenever a full explanation of the chemistry of lead smelting in the reverberatory furnace has been attempted.

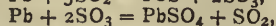
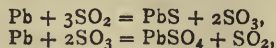
Mr. Hannay had sought (CHEMICAL NEWS, vol. lxx., 1894, pp. 43 to 45) to explain these anomalies by the assumption that a volatile body, PbS_2O_2 , had been formed. He based the evidence for the existence of this body, that could not be isolated, on the observation that in some experiments he had conducted, by passing air over heated galena, he only obtained one-half of the lead in the metallic state according to an equation—



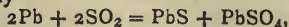
The authors, on repeating this experiment with air and with oxygen, find that the amount of lead that is volatilised in such an operation does not bear any definite ratio to the total amount of lead present, but can be made to vary greatly, and is entirely dependent upon the velocity of the current of air employed. They, therefore, submit that there is now no evidence of any such reaction, the ratio found by Mr. Hannay being the result of some accidental coincidence of experimental conditions.

In continuing the research, the authors found that, on heating a mixture of lead sulphide and lead sulphate in a vacuum, a reaction occurred having sulphur dioxide as one of the products; they also found that the amount of residual lead sulphide and oxide was dependent on the duration of the experiment as well as on the temperatures. They then proceeded to test separately the pairs of bodies supposed to be present, with a view to discover whether secondary or reversed reactions occurred. On heating lead with lead sulphate, they always obtained lead sulphide in the product, in amounts varying with the temperature of the experiment and the length of time that it occupied.

On heating together lead and sulphur dioxide, they found that at moderately elevated temperatures— 300° to 400° C.—lead sulphide and sulphuric anhydride are formed in each other's presence, and that this leads to the formation first of lead oxide and then of lead sulphate. The action is represented more or less completely by the equation—



or more simply—

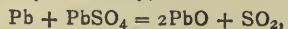


the main condition determining the relative amounts of the products being the temperature at which the experiment is performed under similar conditions.

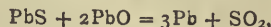
One of the authors is continuing the investigation of

the exact conditions of the equilibrium, as well as its extension to other metals.

The last of these equations occurring in the presence of excess of sulphur dioxide is the exact inverse of one of the main reactions of lead smelting, as stated by Dr. Percy, and in which reaction a supply of air is required to sweep away the sulphur dioxide as soon as it is liberated. The authors submit that, as the new equations quite account for the anomalies to which attention has been drawn, there is now no reason whatever to doubt that the reactions given by Dr. Percy,—



as well as—



do actually take place, and form the basis of the metallurgy of lead.

THE ESTIMATION OF CARBON IN FERRO-CHROME.

By H. BREARLEY and R. L. LEFFLER, F.C.S.

WHEN, three or four years ago, the need to estimate the percentage of carbon in a sample of ferro-chrome arose, the method usually adopted for silica spiegels, and such alloys as are unaffected upon by copper solutions,—viz., combustion of the powdered sample with CuO ,—was resorted to. The results obtained were low, very low. In a neighbouring laboratory it was customary to grind the ferro-chrome and CuO together, burn, re-grind, and burn again. The tediousness of the method, together with the errors of weighing and re-weighing, would make the method an unreliable one in case the alloy was completely decarbonised. But with such heat as we could get, from an ordinary Bunsen combustion furnace, only about 50 per cent of the contained carbon could be extracted after grinding and re-grinding six or seven times. By substituting lead chromate for copper oxide, and prolonging the heat for two hours or so, much better results were obtained. In fact, by re-grinding and re-burning three or four times, nearly the whole of the carbon was eliminated. But this method would not serve when samples were regularly received.

As many periodicals and text-books as were available were searched for a less tedious and more accurate method. A method by Fresenius and Hintz (CHEM. NEWS, lxi., 65)—treatment with chlorine gas and combustion of the carbonaceous residue—stood solitary. Later, Arnold ("Steel Works Anal.," p. 213) published a method which will be referred to later on. Enquiries were made from works' chemists and laboratory instructors as to their respective *modus operandi*, but all either declared their lack of a satisfactory method or preferred to say nothing of the one they had. The following experiments represent the attempts made, at intervals during the last few years, to find an accurate method giving consistent results with as little trouble as possible.

Throughout the ferro-chrome used had been ground to a fine powder in the agate mortar. Leffler shows below that this excessive fineness is sometimes unnecessary.

Of the variable results obtained by simply heating, those were highest which had been most highly heated. This naturally suggested raising the temperature beyond that attainable with the ordinary furnace arrangement. The part of the tube, therefore, containing the porcelain boat was made hotter by means of a blowpipe flame urged by a foot-bellows. The results were higher, more consistent, and more readily obtained.

For clearness sake the method had better be briefly described:—Half to 1 grm. finely-powdered ferro-chrome mixed with 10 grms. lead chromate, which had been previously fused and re-ground to a powder: place in

* Abstract of Paper read before the Chemical Society, May 6, 1897.

porcelain boat and burn in porcelain combustion-tube packed with CuO , &c. In principle there is of course nothing new in this method; it is rather in the matter of accessories and manipulation that the advantage lies.

When the tube is heated by the blowpipe, the mixture in the boat melts and evolves carbon dioxide, sometimes so vigorously as to spirt over the side or strike the roof of the tube. Even if the boat be withdrawn before cooling, the tube will rarely bear re-heating. A few tubes lost this way led to the adoption of glass tubes. No simple tube obtainable would stand the necessary heat, and many varieties—including Schott's Jena glass—were tried. The tubes were covered with asbestos millboard, 1-32nd of an inch or so thick. This plan is adopted with porcelain tubes for ordinary combustions. There is much less danger of fracture on account of sudden heating, and in the case of the best Bohemian glass tubes finally selected it preserved them from bending when sustained at an almost white heat. Over the part which had been strongly heated the surface was pitted with small holes. In places the asbestos was fused into the glass. If the tube was otherwise preserved it invariably cracked on re-heating, on account of this asbestos, so that either the boat had to be changed with the tube at red heat, or a strip of paper had to be wound round the tube before laying over the wetted asbestos. The paper burned, of course, but left a protective covering of ash.

To prevent the boat sticking to the inside of the tube it was covered with a thin piece of millboard and a thicker piece lined the inside, so that the fusion might be removed and the boat preserved. A cover shaped so—

was laid on the top to prevent the spirting melt striking the roof of the tube. It is made to bend over the end of the boat, so that it may not easily be brushed off in passing along the tube. The boat with its fittings is strongly ignited before use.

The lead chromate and sample were mixed by shaking together in a weighing tube. The long shape with wide-stoppered neck is most convenient. A little asbestos on the finger-tip removes the last traces cleanly and effectively.

When the blowpipe has raised the temperature of the tube the gas bubbles through the potash bulb more and more quickly, until the bubbles can scarcely be distinguished. When only half a gram. of the sample is taken, this can be fairly well controlled by judicious heating; but with larger amounts—say from 1 to $1\frac{1}{2}$ grms.—the evolution of gas is beyond control. On unfortunate occasions it is almost vigorous enough to shatter the bulb. After the storm comes the calm. The contents of the boat now greedily absorb oxygen, and unless the supply be quickened the KHO may be pulled back; the soft glass tube will certainly be crushed on to the boat, wrapping it completely round.

The most satisfactory way of showing that the method used was a reliable one was to repeat assays with varying weights of the ferro-chrome.

Two pieces of apparatus, shown in figs. 1 and 2, were used to control the rush of evolved gases. Fig. 1 was used most largely, because it was to hand and already fitted up. It is indeed Stead's "Gas Sampler," and is fully described in Mawson and Swan's Catalogue. One of the lower laps should be replaced by a larger one. Fig. 2 works automatically.

Let the tube be heated by the furnace in the ordinary way, then heat the locality of the boat with the blowpipe. The reaction runs the length of the boat like a train of gunpowder. The "controller" being attached as shown, and the mercury arranged as though a sample of gas was being taken, J and K are opened. With the hand on L, the potash bulb is watched. As the bubbling becomes more and more rapid the O is shut off, the tap (L) suitably turned, and the rush accommodated in G. (A plug of asbestos fibre should have been pushed in after the

boat, to prevent a cloud of lead oxide being drawn along the tube and into the "controller"). The oxygen is re-passed, so as to supply the vigorous absorption in the boat and keep a gentle stream through the bulb. When the absorption in the boat is finished, the gases are forced from G by turning the taps and raising H. The connections are swept out by opening M.

There is no reason why a porcelain tube should not be used over and over again if the noted precautions are taken. The best glass tubes only serve three or four times.

The operation is not so long as the description might suggest. With samples powdered and tubes already

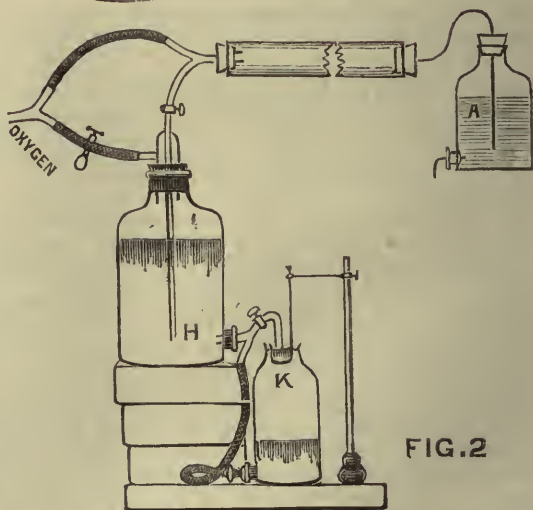
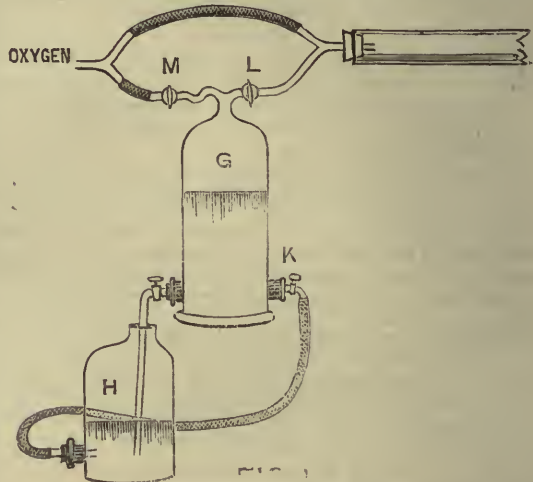


FIG. 2

packed, four samples have been weighed, mixed, boated, and burned in five hours. Such arrangements, partly existing in most metallurgical laboratories, we have used for nearly two years.

We are induced to notice fig. 2 as an alternative to fig. 1, in the hope that it may serve some other purpose. Neither are needed in the operations to be described later. It is necessary to note that the combustion tube, &c., is aspirated by an arrangement indicated at A (fully described CHEM. NEWS, lxxiv., 225), which ensures constant pressure. The two bottles and tubes figured are in principle precisely the same. The small mercury cup, K, prevents any back rush of air, and serves to make the final adjustment of pressures. The furnace is heated,

oxygen passed, and aspirator attached as usual. By means of *K*, or its in-dipping tube, the aspiration due to the fall of mercury is made, so that gas, from the combustion-tube, is just on the point of being drawn through *K*. Any increase of pressure in the tube, such as would be caused by the reaction in the boat, causes mercury to flow from *K*, and provides itself with room in the upper bottle. The gases are re-passed through the combustion-tube by admitting oxygen and running the mercury below the level of *H*, and then sweeping the whole out by passing successively oxygen and air.

The following assays were made with lead chromate, as described on a sample marked 10,543, which contained 61 per cent of chromium. Those marked with an asterisk were done a year ago when the sample was received. The rest were done for this paper.

	I.	II.
0.75 grm. taken.	8.80*	8.86* per cent.
0.75 "	8.84	" "
1.00 "	8.86*	8.83 "
1.25 "	8.90	" "
1.50 "	8.91	" "

When copper oxide is substituted for lead chromate more heat is needed; there is less spirting, and not so violent a reaction. The fusion has the appearance of a piece of gas coke. The general phenomena are similar to those noticed with chromate.

Results on same sample (10,543) are:—

0.75 grm. taken.	8.89 per cent.
1.00 "	8.89 "
1.25 "	8.80 "

Other metallic oxides were tried, more from curiosity than from hope of finding more suitable reagents than copper oxide or lead chromate. In 1859 a patent was taken out for the production of malleable iron by heating with zinc oxide, so long as the iron contained appreciable amounts of carbon metallic zinc, distilled over but stopped when the decarbonisation was complete. With this recommendation zinc oxide was tried, but it could not be made to decarbonise the ferro-chrome within reasonable time at such temperature as could be obtained with our blowpipe.

It need not be argued that it would be an advantage if the combustion could be completed at ordinary furnace temperatures, and in a becoming manner, without the use of any rush controlling arrangement. The use of lead peroxide makes this possible. In this operation the boat need not be covered with asbestos on the inside. The charged boat is placed in the cool combustion-tube. The taps are burned separately and slowly, so as to gradually liberate the oxygen from the PbO_2 . The mixture fuses, and there is the same need to quicken the oxygen to supply the absorption in the boat, but all else goes on with comparative quietness and at ordinary temperatures. Some results on 10,543 with 10 grms. PbO_2 are:—

0.75 grm. taken.	8.87 per cent.
1.00 "	8.86 "
1.25 "	8.87 "

This method we prefer no less on account of accuracy than convenience.

Lead protoxide did not give satisfactory results. In all cases a blank combustion had to be made, but with protoxide the blank was very large and very variable. The sample used was made from the peroxide, but gave a much larger blank, which is most likely due to its well-known property of absorbing carbon dioxide from the air. Very much the same things may be said of the fused variety litharge; although, probably a coincidence, two very good results were obtained.

I have only a mere acquaintance with other methods than those described. Volatilisation in a stream of chlorine, and subsequent combustion of the carbonaceous residue, does not seem to have found much favour. This may be partly due to the fact that the apparatus for such an operation is not in frequent use in steel works' laboratories, and whatever virtue the method may have is more than counterbalanced by the inconvenience of putting things out of joint for a day or two.

Professor Arnold's method has been largely worked by Leffler, who also kindly furnishes other useful information.

The method recommended by Prof. Arnold consists in placing the sample, intimately mixed with lead chromate, in a glass combustion tube between asbestos plugs, and subjecting to prolonged heating in a stream of moist oxygen. The method has been repeatedly tried in asbestos-covered tubes; no naked tube obtainable would stand the ordinary furnace heat, and, unfortunately, Prof. Arnold limits himself to "very hard glass" tubes. Samples kept at a bright red heat from two to four and a half hours only yielded about fifty per cent of the total carbon obtainable. (Found, 4.44, 4.57, 4.77, on a sample containing 8.69 per cent C). But if the temperature of this arrangement was raised by the blowpipe, an additional amount of carbon, bringing the results up to the known percentage, was obtained. Occasionally the oxide of lead formed acted severely on the glass. A current of moist oxygen seemed to have no more effect than a dry one; and, theoretically at least, it seems to be a disadvantage in this way. The oxide of lead formed by the reaction between the reduced lead and oxygen might conceivably be—and in some cases, indeed, was—carried to the far end of the tube. These traces, if dry, may absorb no appreciable amount of CO_2 ; but the hydrate of lead protoxide, made possible by the deposited moisture, very readily absorbs that gas.

It is generally stated that the ferro-chrome used should be in the finest possible state of division. Grinding in the agate mortar is a painful job, and the ease with which some of the reagents mentioned above decomposed the ferro-chrome suggested that this state of "impalpable flour" was sometimes unnecessary. A sample was therefore prepared in three states of division.

1. Passing through wire sieve of 30 meshes to the inch but not through 60.
2. Through 60 but not 90.
3. Through a 90.

The sample contained 8.69 per cent C.

Sample through—	Per cent of carbon with—		
	$PbCrO_4$.	CuO .	PbO_2 .
30 not 60	3.76	1.02	7.58
60 " 90	8.60	8.68	8.67

In the 30, with $PbCrO_4$ and CuO , the grains of the ferro-chrome were distinctly visible after the combustion; the CuO , too, was not even fritted. With this comparative coarseness of sample, the reaction was noticeably less violent. It was not considered necessary to make assays on the 90 after obtaining such accurate results as are shown with 60. Of course, the assays with $PbCrO_4$ and CuO were blasted, and all of them carried out as previously mentioned. The following are examples showing that the carbon percentage is very variable:—

C.	Cu.
5.82	89.46
8.40	60.02
9.20	60.73
9.39	61.84
10.18	60.25
11.22	67.24

The Laboratory,
Messrs. Thos. Firth and Sons, Lim.,
Sheffield.

CONTRIBUTION TO THE STUDY OF
HYPONITROUS ACID.*

By A. HAUTZSCH and A. L. KAUFMANN.

(Continued from p. 233).

Free Hyponitrous Acid.

HYPONITROUS acid was first obtained in the free state in aqueous solution by Van der Plaats (*Berichte*, vol. x., p. 1507). It has also been studied by Thum (Wiener, *Monatsh.*, vol. xiv., p. 294). About the same time as we did, Tanatar obtained it in an oily state, probably still containing water. To obtain this acid in the solid state it is necessary to proceed as follows:—A current of hydrochloric acid gas, dried with the greatest care, is passed through ether absolutely free from water. To this etherised hydrochloric acid, perfectly dry hyponitrite of silver is added little by little. The solution must be kept cool, and special precautions must be taken to prevent the access of moisture. Hyponitrite of silver is added until the last portion added no longer loses its colour; that is to say, until the etherised solution no longer contains any free hydrochloric acid. This solution is then rapidly filtered on a dry filter, and placed in an exsiccator *in vacuo*. Moisture and acid fumes must be guarded against with the utmost care, and for this reason a crucible containing solid caustic potash should be placed in the exsiccator. The evaporation of the ether can be accelerated by passing through the etherised solution a current of air which has been first passed through two wash bottles—the first containing a solution of caustic potash, the second sulphuric acid—and then through a tube containing phosphoric anhydride. It is advisable to keep the exsiccator in a refrigerating mixture; the ether then evaporates, leaving the hyponitrous acid in the form of white scales.

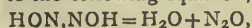
Hyponitrous acid is very explosive. When absolutely dry it decomposes spontaneously; the decomposition is accelerated by the presence of acid fumes, but retarded by cold. Simply touching it with a glass rod is enough to cause its decomposition. Contact with solid caustic potash is sufficient to cause sudden decomposition accompanied by ignition.

Hyponitrous acid is not only very soluble in water, but it also dissolves with great readiness in alcohol, ether, chloroform, and benzene.

We have not been able to carry out the analysis of this acid. So long as there is any ether present, the crystals can be preserved for some time, but as soon as they become quite dry, they detonate even at 6° without any apparent cause.

We have, however, been able to show, by qualitative reactions, that the substance obtained was really hyponitrous acid. We placed some of the crystals on a plate of porous porcelain, and the moment they became dry they were slid off into a watch-glass containing iced water. The solution gave, on the addition of nitrate of silver, the characteristic hyponitrite of silver. The aqueous residue obtained by the decomposition of the crystals also gives the same reaction.

The water formed by the decomposition of hyponitrous acid, according to the following equation,—



dissolves a portion of the acid, and thus saves it from decomposition.

In aqueous solution, hyponitrous acid is much more stable than in the anhydrous state. Although, as will be shown further on, the acid does decompose rapidly enough when in aqueous solution at 25°, it is still sufficiently stable at 0° to enable one to determine its molecular weight by the cryoscopic method.

Determination of the Molecular Weight.—For this operation we used pure water, whose freezing-point had

been determined in Beckmann's apparatus, and with it we prepared a one-sixth normal solution of hydrochloric acid, by passing through it a current of hydrochloric acid gas and diluting to the proper point. 200 c.c. of this solution, which contained 0.1217 grm. of HCl, lowered the freezing-point by 0.625° (mean of five experiments). Without removing the Beckmann apparatus from the freezing mixture, we added a solution of hyponitrite in excess. The one-sixth normal solution thus obtained (0.1035 grm. in 20 c.c. of water) produced a lowering of the freezing-point of only 0.167° (mean of three observations). The lowering of the congelation-point produced by equivalent quantities of hydrochloric acid and hyponitrous acid, are in the proportion of 1 to 3.8. As at this degree of dilution (the molecular quantity in 6000 litres of water) hydrochloric acid is completely dissociated, and has a molecular weight of 18.4, and a number of ions = 2; the substitution of $\text{H}_2\text{N}_2\text{O}_2$ for HCl ($2\text{H} + 2\text{Cl}$) ought to result in the reduction of the number of molecules to one quarter. This is only possible if the hyponitrous acid, very much diluted (molecular quantity in 3000 litres of water), does not appreciably dissociate itself into its ions; that is to say, if it is a very weak acid, that which our experiments on its conductivity fully confirm. We have then:—Molecular weight: found, 59; theory, $\text{H}_2\text{N}_2\text{O}_2$, 62. This result proves that, under the conditions we have just indicated, hyponitrous acid does not exist in the state of a simple molecule—



Action of Alkalis.—If hyponitrous acid closely resembles carbonic acid by its ready dissociability, the analogy between these two acids is shown in a still more striking manner in their titration by means of alkaline solutions, using phenolphthalein as indicator. Thus, as Thum has already shown, the titration indicates only half the amount of acid present. The following experiment leaves no doubt on the subject:—

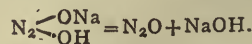
To 20 c.c. of a decinormal solution of hydrochloric acid hyponitrite of silver was added in excess. The hyponitrous acid liberated was immediately neutralised at 0° by a titrated solution of baryta, using phenolphthalein as indicator.

	Theory.	
	BaN_2O_2 .	$\text{BaH}_2\text{N}_2\text{O}_2$.
Solution of baryta used, 5 c.c. 50	10 c.c. 9	3 c.c. 45

Monosodic hyponitrite, like monosodic carbonate, is neutral to phenolphthalein. Thus, this analogy exists:—



Further, acid hyponitrite is not stable in aqueous solution. If, in a solution of hyponitrous acid titrated with an alkaline solution until the red colouration of phenolphthalein shows, we cause the colour to disappear by adding a drop of hydrochloric acid, and let it stand, the red colouration will reappear. The acid hyponitrite of soda decomposes very slowly into protoxide of nitrogen and caustic soda, according to the equation—



We can follow this decomposition by determining the increase of the alkalinity of the solution. Thus, for example, in one experiment 43 per cent of the amount of hyponitrous acid which was originally present in the solution in the state of an acid salt was decomposed after eighteen hours at the ordinary temperature.

Reactions of Hyponitrous Acid.—Contrary to the assertion of Van der Plaats, solutions of hyponitrous acid do not decompose iodate of potassium, liberating iodine (Thum). We found that on adding to a freshly prepared solution of free hyponitrous acid a solution of potassic

* *Moniteur Scientifique*, vol. xi., p. 336, May, 1897.

iodide and starch, slightly acidulated with acetic acid, the blue colour does not appear at first, but after the lapse of some time the liquid becomes blue, and the colour continues to become more and more intense. A further remarkable thing is, that on adding hyponitrite of silver to cold concentrated sulphuric acid, and adding a little diphenylamine, a very intense blue colouration is produced. With a solution of ferrous sulphate a brown ring is obtained. These reactions are characteristic of nitrous and nitric acids. We shall refer to them again later on, when discussing the decomposition of hyponitrous acid.

Hyponitrite of Ammonia, $\text{H}_4\text{NOH.NHO}$.—This salt was hitherto unknown. Zorn observed that the solution obtained by the reaction of hyponitrite of silver on a solution of chloride of ammonium, which should contain hyponitrite of ammonium, decomposed very rapidly at the ordinary temperature, with evolution of gas. However, it is possible to isolate an ammoniacal salt by passing a current of well-dried ammonia gas through an etheric solution of hyponitrous acid, as already described. After a very short time the salt is precipitated in the form of a white mass. It is filtered rapidly, washed with ether, and dried on plates of porous porcelain.

Hyponitrite of ammonium melts about 64° to 65° , decomposing with violence. It is easily soluble in water, giving an alkaline reaction. But it has not been recovered either from its aqueous or alcoholic solutions, even when working *in vacuo*. The solutions leave no residue whatever on evaporation. Jackson's assertion (*Berichte*, vol. xxvii., R., p. 562) that hyponitrite of ammonium could be obtained in beautiful crystals by the reaction of hyponitrite of silver on an alcoholic solution of sulphide of ammonium is evidently founded on an error. In the solid state this substance decomposes slowly at ordinary temperatures into ammonia, water, and protoxide of nitrogen, and therefore cannot be brought to a constant weight. It is for this reason that exact analytical results cannot be obtained. The ammonia has been estimated by decomposing the freshly-prepared and rapidly weighed salt (taking no note of the loss of weight) by caustic potash, absorbing the gas in a normal solution of sulphuric acid, and titrating the excess of acid. Three estimations carried out on three portions of one and the same sample gave the following results:—

Grm.	Per cent.
I. 0.0560 of hyponitrite of ammonia gave 20.34 NH_3 .	
II. 0.0947 " " " 20.10 "	
III. 0.0631 " " " 19.94 "	

Theory:—

$\text{NH}_4\text{HN}_2\text{O}_2$..	25.52 per cent
$(\text{NH}_4)_2\text{N}_2\text{O}_2$..	35.42 "

We did not continue our experiments on the estimation of the protoxide of nitrogen by decomposing the salt in the endiometer with concentrated sulphuric acid. Our first attempt resulted in a violent explosion, which blew the apparatus all to pieces.

The spontaneous decomposition of hyponitrite of ammonia was studied by determining the loss of weight that the salt sustained when left in the exsiccator *in vacuo*. The following are some of the results obtained:—

Time. Hours.	Weight. Grm.	Loss of weight. Per cent.
0	0.01057	—
87	0.0214	79.75
119	0.0169	84.0
167	0.0136	87.13

This decomposition of acid hyponitrite of ammonia resembles that of bicarbonate of ammonia, only the latter is more stable. We have not succeeded in preparing the neutral hyponitrite of ammonia, even when passing a current of ammonia through hyponitrous acid for a considerable time. Here, again, the analogy existing between

hyponitrous acid and carbonic acid is evident; for, as is well known, the neutral carbonate of ammonia is rapidly transformed into acid carbonate when exposed to the air.

(To be continued).

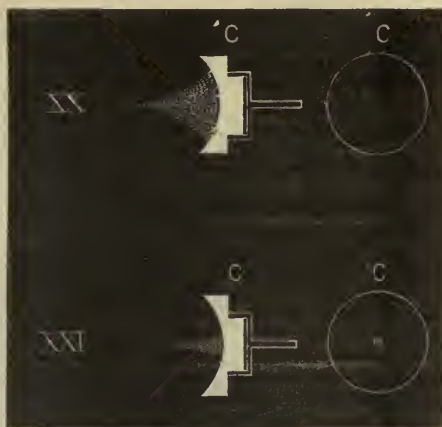
SOME EXPERIMENTS WITH CATHODE RAYS.*

By A. C. C. SWINTON.

(Concluded from [p. 236].)

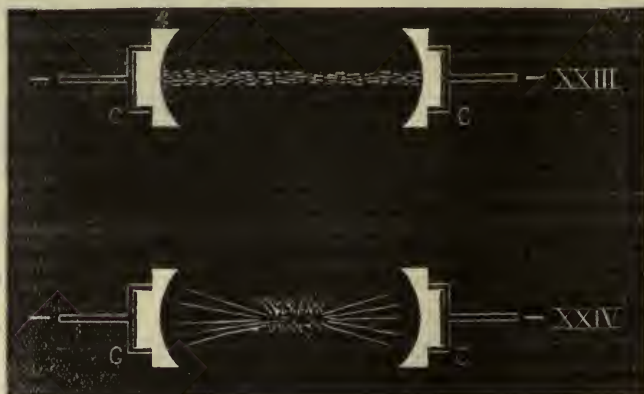
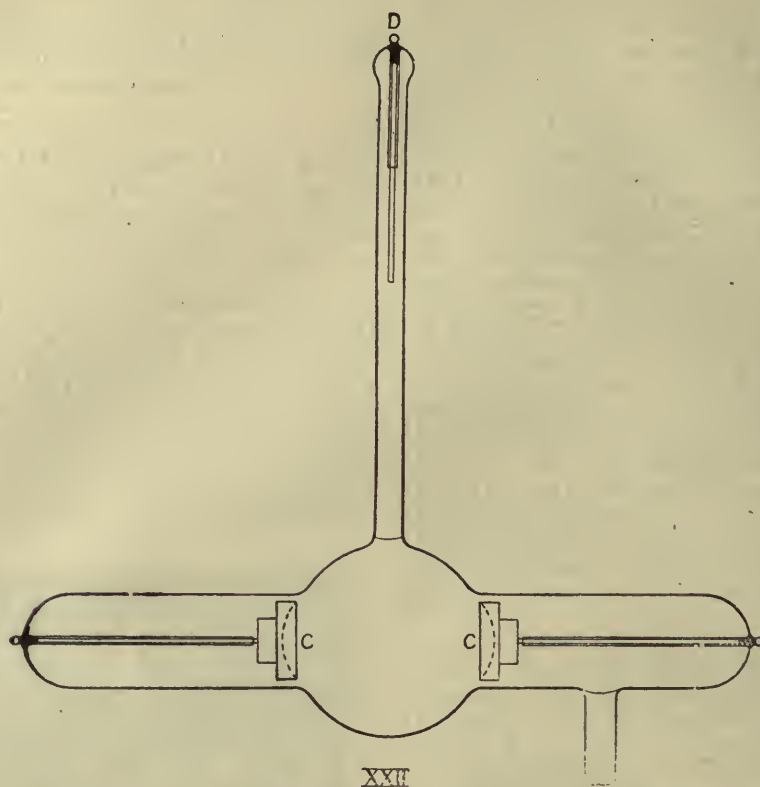
The Convergent Cone at Higher Vacuo.

As has been mentioned, the carbon anti-cathode screen was found useless for investigating the convergent cone of cathode rays at anything but a very low vacuum, by the reason of the well-known difficulty in getting any discharge to pass when the distance between the electrodes is less than the thickness of the dark space, and for the further reason that if the anti-cathode screen was not connected to the anode, it became itself negatively charged and acted as an additional cathode when brought into the space between the cathode and the focus.



Under these circumstances it was thought that possibly some additional information might be obtained with regard to the form of the convergent cone at high vacua, by making the concave cathode itself of carbon. A tube was therefore constructed having a concave carbon cathode, the diameter of which was 1 inch, and the radius of curvature 0.75 inch. The appearance of the cathode with this tube is shown for a fairly high vacuum in fig. 20, in which the cathode itself is shown in section, so as to let the form of the discharge be better seen. As will be observed under this condition of vacuum, which was too high to show any divergent cone, the cone of convergent rays appears to be contracted in diameter at its base, and to come off from the central portion of the cathode only, the remaining surface of the cathode being apparently inactive. This was found to be still more the case at higher vacua, as will be seen from fig. 21, which shows in a similar manner the form of the cathode discharge in a tube exhausted to a very high vacuum. In this case, as will be observed, the whole of the cathode rays appear to come off from a very small spot in the centre of the cathode. Further, that this small spot is, at any rate, the source of most, if not all, activity, was evident from the fact that it became luminescent exactly in the same manner, but in a less degree, than had previously been

* A Paper read before the Royal Society, March 11, 1897.



observed with a carbon surface upon which cathode rays were concentrated. Whether this surface luminescence of the cathode carbon at the point where the cathode rays leave it is due to the violent tearing away of particles of carbon, or to some other cause, it is difficult to say, but the fact that at high vacua the cathode rays come off entirely, or at any rate almost entirely, from only a very small portion of the centre of the cathode, explains the observed fact that within limits large cathodes have no advantage over small cathodes in X-ray tubes.

During the carrying out of the above experiments with a carbon cathode, very bright sparks were occasionally seen coming off the cathode and passing through the focus, and it was consequently thought that possibly by placing two concave carbon cathodes facing one another, such particles, by being caused to rebound backwards and

forwards continuously between the two, might render the form of cathode stream visible at very high vacua when the stream itself becomes otherwise invisible.

With this view a tube was made as shown in fig. 22, in which two concave carbon cathodes, C C, similar to those employed in the last experiment, were placed exactly opposite one another, so that a prolongation of the focus of either one would pass through the centre of the other. The anode, D, was placed in an annex, as shown in the illustration, and the two cathodes were connected together by means of a wire outside the tube. At a very high exhaustion this tube gave very beautiful effects, and showed clearly the form of the cathode discharge at a degree of exhaustion when it is usually in itself quite invisible. Immediately on the current being turned on and the discharge passing, a straight and thin stream of bright golden

coloured particles, of apparently incandescent carbon, passed between small luminescent spots at the centres of each cathode, as shown in fig. 23. This did not last for more than a second, when, owing no doubt to the rapid fall of vacuum, the appearance changed to that shown in fig. 24, and the incandescent particles of carbon could be seen passing backwards and forwards along the convergent and divergent cones of cathode rays, which, at the lower vacuum, proceeded from both cathodes, and spluttering in the centre, where the particles going in opposite directions collided. This appearance lasted for some seconds, becoming gradually fainter as the vacuum fell. By re-exhausting the tube with the pump, however, the original appearance shown in fig. 23, as also the appearance shown in fig. 24, could be produced as often as desired. Apparently the particles of carbon become heated to incandescence either by the action of the cathode rays upon them while they are flying through space, or by their friction in passing through the residual gas, and possibly by their mutual collisions, for in the stage shown in fig. 24, when the cathodes themselves show no luminescence, the flying particles appear to be most intensely luminescent when in the centre of the tube. It may be mentioned that after this experiment had been repeated several times, the glass of the tube became perceptibly blackened, which, taken with the fact that a similar tube with cathodes of aluminium showed no stream of bright particles, goes to show that the particles consist of carbon torn off the surfaces of the cathodes.

The Production of X-rays.

The tube, fig. 22, with carbon cathodes was found to produce feeble X-rays, which, when observed with a fluorescent screen, appeared to come either from the fluorescent glass of the bulb or from the travelling particles of carbon.

In order to ascertain whether it is necessary that the cathode rays should fall on solid matter in order to produce X-rays, another tube was constructed, similar in all respects to that shown in fig. 22, with the exception that the two cathodes were made of aluminium.

It was thought that with this tube the opposing streams of cathode rays might possibly produce X-rays at the point where they met. This does not however, appear to be the case, as though this tube, when exhausted to so high an extent that the alternative spark in air leapt fully 8 inches, gave X-rays in considerable quantity, these rays appeared to come entirely from portions of the glass of the tube that were covered with green fluorescence, and not at any rate appreciably from the central point between the two cathodes, where the opposing streams of cathode rays would meet one another.

It seems, therefore, that X-rays can only be produced by cathode rays when these strike solid matter.

In conclusion I wish to mention how much I owe in carrying out these experiments to the assistance of Mr. J. C. M. Stanton and Mr. H. Tyson Wolff, who have made and exhausted all the tubes, and to whom I am also indebted for many valuable suggestions.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING APRIL 30TH, 1897.

By WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolitan Water Act, 1871.

London, May 10th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 168 samples

of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from April 1st to April 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in a previous report.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 168 samples examined all were recorded as clear, bright, and well filtered.

The rainfall at Oxford during the month was 1.95 inches; the average for 30 years is 1.66 inches, making an excess of 0.29 inch; 0.54 inch fell on the 21st, the rest was fairly evenly distributed throughout the month. We have now had a total excess of 2.02 inches this year on an actual fall of 8.82 inches, or nearly 25 per cent.

Our bacteriological examinations of 234 samples give the following results:—

	Microbes per c.c.
Thames water, unfiltered (mean of 24 samples)	3277
Thames water, from the clear water wells of five Thames-derived supplies (mean of 118 samples)	38
Ditto ditto highest	335
Ditto ditto lowest	1
New River, unfiltered (mean of 23 samples) ..	892
New River, filtered (mean of 23 samples) ..	13
River Lea, unfiltered (mean of 23 samples) ..	708
River Lea, from the clear water well of the East London Water Company (mean of 23 samples)	27

These figures give a sufficient proof of the efficiency of the filtering beds and the excellent quality of the water supplied to the Metropolis.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.
JAMES DEWAR.

THE QUANTITATIVE AND QUALITATIVE SEPARATION OF BARIUM, STRONTIUM, AND CALCIUM.*

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In the quantitative or qualitative separation of barium, strontium, and calcium, there has been always a considerable amount of difficulty due to the remarkable properties of strontium which in so many respects lie almost midway between those of barium and of calcium. Hence though the estimation of both barium and calcium when present together, or of strontium when alone, presents but little trouble, yet if the whole three members of the group be taken the task becomes one of greatly-increased difficulty, it being no easy matter to ensure either the complete precipitation or the entire retention in solution, according to the process to be employed, of the strontium. To attain one or other of these ends many methods have been proposed; among these I may mention the following:—Fresenius recommends in his work the precipitation of the

* A Paper read before the Society of Chemical Industry (Yorkshire Section), January 25th, 1897. From the *Journal of the Society of Chemical Industry*, vol. xvi., No. 2.

barium as silicofluoride and the subsequent separation of the strontium and calcium by means of prolonged boiling with an enormous excess of ammonium sulphate. The particulars of the remaining processes are taken from the *Journal of the Society of Chemical Industry*. Fresenius and Ruppert (1892, p. 776) have investigated the relative solubilities of strontium and calcium chromates in dilute alcohol; though of value qualitatively they consider that quantitatively the method is lacking in accuracy. McElroy and Biglow (1893, p. 181) suggest the use of aqueous acetone upon these same salts, but seemingly only for qualitative purposes. In this year also (1893, p. 627) Fresenius recommends the separation of calcium as nitrate from the other nitrates by its solubility in ether-alcohol, the results he gives being very accurate. Browning (1894, p. 282) carefully examined the solubility of strontium nitrate in boiling amyl alcohol, in which the salt is almost insoluble; the corresponding calcium nitrate dissolves readily, but to effect a complete separation a second treatment is necessary. Dupasquier (1895, p. 822) points out that calcium is converted into a tartrate by boiling in a liquid containing a soluble tartrate and sulphate, while barium and strontium remain as sulphates. These are estimated indirectly for quantitative purposes. Baubigny (1895, p. 1065) separates the strontium as sulphate in the presence of potassium sulphate, but the precipitation is not complete.

This bibliography, though not complete, includes the most important of the plans suggested. It will be noticed that in almost all of them there arises the difficulty of the slight solubility of the strontium salts in the solvents necessarily employed. Further, it rarely happens that the same method can be used both quantitatively and qualitatively. But the special objections to these methods are due to the fact that either out-of-the-way reagents are employed, or that much and prolonged boiling is necessary to convert the given salts into the required modifications. Both these are hindrances in the way of quantitative analysis, and are most serious detriments to qualitative work.

The principle of the separation upon which I have worked depends entirely upon the behaviour of the nitrates of these three metals towards concentrated nitric acid. The method is also readily applicable to qualitative analysis; the nitrates of barium and of strontium are quite insoluble in this acid, whilst calcium nitrate dissolves very rapidly. That barium nitrate is insoluble appears to be accepted, but there is considerable divergence of opinion as regards the strontium salt; thus, Wurtz (*Am. J. Sci.*, [2], xxv., 377) states that it is sparingly soluble in concentrated nitric acid, whilst Schultz (*Zeit. Ch.*, [2], v., 537) considers it to be insoluble. The experiments by which I sought to settle this initial contradiction were as follows:—The nitric acid used, and which was bought as pure, was re-distilled, the water also being similarly treated. In the case of both the nitric acid and of the water, portions upon evaporation either left no residue or one which it was not possible to weigh. The carbonate was obtained, at different times, from Germany, and as being perfectly pure. I may here mention that it is most difficult to procure pure strontium carbonate. All the material which I obtained invariably contained traces of calcium in amounts varying from 0.25 per cent to even 2 per cent. The presence of this impurity caused me great inconvenience, and at first much loss of time. In order to purify the carbonate a considerable quantity was converted into nitrate and thoroughly stirred with nitric acid, filtered, and the residue dissolved in water and precipitated as carbonate, washed, and dried. In the spectroscopic no trace of the calcium lines were now visible. So satisfactory and rapid is this method that I would recommend it strongly as being the most easy and the most safe for the purification of strontium from calcium salts. I may here mention that throughout my experiments I always used the carbonates of the metals partly because of the much greater convenience and certainty in weighing, and also because

the carbonates are far more likely to be required for estimation under usual conditions than the nitrates, and therefore it seemed advisable to work upon the more commonly occurring body.

The pure carbonate was treated with dilute nitric acid and evaporated to dryness upon the water-bath. Pure concentrated nitric acid (sp. gr. 1.445) was then added and the mixture kept well stirred for many hours, in some cases three days. The solution was then filtered through filter-paper or glass-wool, and the filtrate evaporated to dryness, taken up with a little hydrochloric acid, and again evaporated. The residue gave, in the spectroscopic, no trace of the strontium lines, but consisted of small traces of sodium and iron sulphates derived from the acids employed, and of celluloid matter arising from the filter-paper, when used. Similar experiments were repeated many times, varying the amounts of carbonate and of nitric acid, but only upon one occasion, in one of my earlier experiments, did I distinguish the strontium lines. I had treated some 3 grms. of the carbonate with nitric acid, and upon filtering and evaporating to dryness the residue clearly contained strontium. This was due to the solution of traces of the nitrate, the nitric acid having become diluted by the considerable amount of water formed in the conversion of the carbonate into nitrate. On all subsequent occasions I invariably evaporated the nitrate to dryness upon the water-bath, and then took up with fresh nitric acid. Under these conditions the reaction was only as between the nitrate and nitric acid, and the strontium lines did not again become visible in the filtrate. Hence strontium nitrate is insoluble in concentrated nitric acid. Direct experiments upon barium nitrate showed this salt to be also insoluble, whilst calcium nitrate readily dissolved. As regards the nitric acid, I prefer the specific gravity to be about 1.46 (corresponding to 80 per cent of the acid) or even higher, but for most purposes the ordinary acid of specific gravity 1.42 (containing 70 per cent of HNO_3) will suffice.

The actual quantitative estimation of these elements carry out in the following manner:—The carbonates are evaporated with nitric acid, either in a beaker or in a porcelain dish, preferably the latter, upon the water-bath until the mass is quite dry. Concentrated nitric acid is added in excess, and the mixture kept well stirred. The crystals settle out after each stirring very rapidly, and the clear supernatant liquid is poured through a double filter-paper which has been moistened previously with concentrated nitric acid. The residue may be washed either with the concentrated acid by decantation, a method to which the mass from its crystalline nature readily lends itself, or in the customary way upon the paper.

The filtrate is evaporated either to dryness, taken up with hydrochloric acid, and the calcium precipitated as oxalate, or evaporated down with sulphuric acid, and the residue after ignition weighed as sulphate.

The remaining nitrates are dissolved in water, and the solution made alkaline with ammonium hydrate, acidified with acetic acid, and the barium precipitated as chromate. The filtrate is warmed with hydrochloric acid and alcohol until the chromate is reduced, the chromium precipitated as hydrate and filtered off. The filtrate is evaporated to dryness upon the water-bath with a little sulphuric acid. The residue is treated with dilute alcohol and washed, and the residual strontium sulphate weighed as such. Two out of the different test analyses which I have made give the following results:—

	1.		2.	
	Taken.	Found.	Taken.	Found.
Barium carbonate ..	0.5393	0.5379	0.2127	0.2130
Strontium carbonate ..	0.2578	0.2584	0.4583	0.4576
Calcium carbonate ..	0.5473	0.5470	0.2773	0.2780

For the qualitative separation the above process may be much simplified. The carbonates, after treating with the minimum amount of nitric acid, are boiled down just to dryness; then treated with concentrated nitric acid, the

paper having been first moistened with this acid, filtered, and the residue washed by decantation. The filtrate is diluted considerably with water made alkaline with ammonia, and the lime thrown down as oxalate. The residue is dissolved in water (the filter-paper will not disintegrate unless boiling cold water be used), made alkaline with ammonia, acidulated with acetic acid, and the barium precipitated as chromate. To the filtrate is added hydrochloric acid, and it is boiled with a little alcohol, and the chromium precipitated as hydrate, which is filtered off. To the filtrate sulphuric acid is added, and the solution heated; if necessary alcohol is added, the strontium coming down as sulphate. The separation of the chromium as hydrate is not in all cases necessary.

The process can be carried out very rapidly, and, further, is a complete separation of these three metals, and does not rest upon grounds such as the length of time allowed for boiling or standing, or the greater or lesser concentration of solutions.

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, May 14th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

MR. W. WATSON described "*An Instrument for comparing Thermometers with a Standard.*"

The thermometers to be compared are inserted together in an enclosed vapour-tube, the temperature of which can be maintained very constant at different parts of the scale. The apparatus is an adaptation of the arrangement devised by Ramsay and Young for vapour densities. It consists of a wide vertical glass tube, with a narrower tube attached at the top. The narrow tube bends downwards and communicates with a closed vessel of considerable volume. A portion of the vertical tube is surrounded by a condensing-jacket, and a manometer tube is inserted near the top. The object of the large vessel is to diminish errors arising from fortuitous changes of pressure resulting from small leakages or "bumping" of the boiling liquid. Electrical heating of the bulb containing the liquid effectually removes the "bumping." The following liquids, used consecutively, give a range of temperature from 20° C. to 120° C.: carbon bisulphide (20°–46°), ethyl alcohol (80°), chlorobenzene (120°). The apparatus, when once started, requires very little attention; from results submitted by the author, the variations do not exceed 0.02° C. per hour. In constructing the various parts, the difficulties of glass-blowing are reduced by making the joints of indiarubber stoppers, attached to the glass with indiarubber solution. Each joint is jacketed with glycerin. If the above liquids are used in the vapouriser, the scales of the thermometers can always be read within the tube; it is only with water that the condensed vapour gives trouble.

Prof. AYRTON thought the apparatus would come into extensive use; it did away with errors arising from differences of length of thermometer stems, it left no question as to the equality of temperature of the two bulbs, and there was no probability of error due to a difference of thermal "lag" in any two thermometers.

Mr. WATSON, in replying to a question of Prof. Perry's, said the fact of using indiarubber joints limited the available range of temperature. Working with blown joints, Ramsay and Young had found no difficulty in their vapour-density experiments at higher temperatures.

Prof. CAREY FOSTER read a paper by Mr. D. K. MORRIS, of Zürich, on "*The Effect of Temperature upon the Magnetic and Electric Properties of Iron.*"

The investigation relates to the measurement of the

magnetic permeability, hysteresis, and electrical resistance of iron simultaneously at different temperatures. The specimens are formed into annular rings made from iron strip. The strip is first lapped round with asbestos-paper and mica, and then wound upon itself to the requisite thickness. A platinum wire is included in the mica lappings, for thermometrical purposes. Upon each annular ring are the following windings:—(1) A primary magnetising coil. (2) A secondary coil connected to a ballistic galvanometer. (3) An electrical heating coil. Further, the iron strip is itself connected to a Wheatstone's bridge, for resistance measurements. The coil can be heated to 1050° C. At the higher temperatures, the surrounding air has to be freed from oxygen; this is done by enclosing the coil in a suitable vessel and exhausting with an air-pump. When most of the air has thus been removed, the residual oxygen is absorbed by an electrically heated iron wire. Curves are drawn representing the changes of permeability at the different temperatures, and, at the same temperature, the corresponding hysteresis loops are plotted. The hysteresis diminishes with temperature; it nearly vanishes at about 764° C.

At the suggestion of Prof. Ayrton, it was agreed that the discussion on this paper should be adjourned until the publication of the results. The paper will therefore be printed without delay.

Mr. ROLLO APPELVARD read a paper on "*The Formation of Mercury Films by an Electrical Process.*"

If a sheet of damp leather, or similar permeable substance, is used as a separating diaphragm between two bodies of mercury, and a current is sent through it, a film of mercury is deposited upon the surface connected to the positive pole; and the film remains on the diaphragm after removal from the apparatus. If the diaphragm is replaced in the apparatus and subjected to a current in the reverse direction, the film vanishes from that surface, and a second film appears on the other side; that is to say, the film is always on the side of the diaphragm connected to the positive pole of the battery, and there is no film on the negative surface. Different diaphragms and films were exhibited—of filter-paper, asbestos-paper, plaster of Paris, &c. A current of about one-fiftieth of an ampère, or more, is necessary. A sheet of tin-foil, included between folds of filter-paper, becomes perforated with pin-holes when the current is passed between the outside surfaces. This happens whether the outside electrodes are mercury or metal plates. If the top electrode should be tin-foil, this also becomes perforated as well as the included sheet. A further experiment was shown, in which a gold coin is placed upon the folds of filter-paper; the current produces a gold discolouration which penetrates the folds. This, it was suggested by the author, may help to account for the formation of metallic lodes and veins as they exist in rocks; and they may partly explain the "inductoscripts" of Mr. F. J. Smith.

Dr. S. P. THOMPSON said he did not know of any other examples of an *anode* being more active—mechanically—than the *cathode*, except the electric arc. He was surprised that the film should appear on the positive surface.

Mr. SHELFORD BIDWELL thought selenium presented, in some of its actions, an example of the *anode* being thus active.

Prof. AYRTON said that if a vessel containing a substratum of mercury amalgam was filled up with water in which gold crushings were washed, the gold descended into the amalgam. This, however, might be partly due to gravity, and partly to simple electrolysis.

Mr. APPELVARD said he had no definite views as to the formation of the films. He believed it to be a secondary effect of electrolysis, aided by electric osmosis. The experiments of Mr. C. K. Falkenstein upon the electric tanning of leather, and the early results of M. Perret, helped the idea of electric osmosis; they were not suffi-

cient, however, to justify that theory without further research. A careful chemical analysis of the deposits left in the folds of filter-paper would be the best guide.

The PRESIDENT proposed votes of thanks to the authors, and the meeting was adjourned until May 28th.

THE ROYAL SOCIETY.

A CONVERSAZIONE was held at the Royal Society's Rooms at Burlington House, on Wednesday, the 19th of May. The guests and Fellows were received by the President, Lord LISTER.

Among the exhibits were some interesting colour-photographs, produced by the Danzac-Chassagne process, the details of which are not known, but the prints (silver) are believed to be treated with a solution containing albumen and certain metallic chlorides, and afterwards with colouring materials—blue, green, and red.

Mr. Campbell Swinton showed a good collection of the various latest forms of X-ray tubes.

In the principal Library, Prof. Roberts-Austen showed his apparatus for micro-photography, and some slides showing the mode of existence of carbon in steel; also a diamond made by himself, by M. Moissan's method—the magnifications vary from 500 to 1000 diameters.

Messrs. C. T. Heycock and F. H. Neville had a curious alloy of silver and zinc, which would have warmed the hearts of the old-time alchemists. At the ordinary temperature it is the colour of silver, but when warmed up to 300° C. and suddenly cooled it becomes copper-coloured or bright red, and remains so; if again heated, and cooled slowly, it resumes its original colour. This is not an effect of oxidation, as the phenomenon occurs equally well in hydrogen gas or in vacuo.

Prof. Oliver Lodge exhibited an apparatus, demonstrating Zeeman's discovery of the broadening of spectrum lines by the action of a magnetic field on the source of light. By reason of reversals, the usual appearance of each sodium line is as if it were double; the magnetic field makes it appear triple, or even quadruple.

Perhaps one of the prettiest exhibits is Prof. Silvanus Thompson's model of a Hertz wave transmission, showing the manner in which a wave can be transmitted to and received by a resonator in tune with the oscillator.

At 10 p.m. Prof. Ayrton gave a demonstration with experiments and lantern slides, on "Some Electric and Mechanical Analogues"; and at 11 p.m. Prof. Farmer showed some slides from micro-photographs illustrating nuclear division in animal and vegetable cells.

NOTICES OF BOOKS.

The Electric Furnace. ("Le Four Electrique"). By HENRI MOISSAN, Membre de l'Institut. Paris: G. Steinheil. 1897. Pp. 376.

(CONCLUDING NOTICE).

THE preparation of simple bodies by means of the Electric Furnace forms, as we mentioned previously, the subject-matter of Chapter III. The high temperature now reached renders possible many chemical changes which have hitherto been unattainable. We know, for instance, the laws of decomposition by heat, of carbonate of lime; but until now carbonate of barium has been considered to be undecomposable, simply on account of the limited means at our disposal. It is now shown that carbonate of barium, like chalk, loses its carbonic acid at a very high temperature, and is converted into caustic baryta. Further, certain oxides—such as silica, the alkaline earths, oxides of uranium, vanadium, and zirconium—have now

been directly reduced by carbon, yielding the pure metals themselves, or metallic carbides.

Some of these highly refractory substances, though reducible to the metallic state, have not yet been produced in the form of ingots, but persist in remaining in a powdery state, a phenomenon analogous to very finely-divided mercury. Calcium, strontium, and barium are among these; on the other hand, chromium, molybdenum, titanium, uranium, &c., can be produced in ingot form without much difficulty. For the purpose of his research on chromium, M. Moissan prepared 40 kilograms of the metal.

The details of the manufacture, the precautions taken in so doing, and the analyses of the samples of ten of these rare and interesting metals are then given.

The general method of procedure will be by now easily understood; but the various and minute details, to be followed in the handling of such large currents, and enormous temperatures, in the production of these chemically speaking simple bodies, afford many pages of most interesting matter.

Alumina has always been regarded as irreducible; but if a perfectly transparent crystal of corundum be placed in a carbon crucible, in a carbon tube, and exposed to the heating effect of a current of 1200 ampères and 80 volts, it is volatilised in a few minutes. The crucible, completely converted into graphite, does not contain a trace of ash, but the surface of the tube on either side is covered with a deposit of crystals of graphite and metallic aluminium in spheres up to 2 or 3 m.m. in diameter. The author has made extensive experiments on the various impurities in commercial aluminium, experiments of considerable importance to the manufacturers of this metal: different samples of aluminium, made by different processes, vary considerably, not only in chemical composition but also in mechanical utility, small quantities of impurities having—as is well known to metallurgists—an enormous influence on the physical properties of many metals. Comparative tests were made with several ingots of aluminium, some containing a small proportion of carbon (different analyses have given 0.104, 0.108, and 0.080 per cent of carbon), and the other containing no carbon. The effect of this minute amount of impurity was very striking; the breaking strain of pure aluminium was 11.1 kilos. per square m.m. and the elongation 9 per cent, while the other samples containing carbon had breaking strains of only 6.5 to 8.6 kilos. per square m.m. and elongations of from 3 to 5 per cent.

Chapter IV. deals with the large class of new compounds known as carbides, borides, and silicides. It would not be correct to say that metallic carbides were unknown before the invention of M. Moissan's furnace. It was known that certain metals would dissolve carbon in variable proportions, generally when the metal was in large excess; but the direct preparation of crystallised carbides was impossible with the furnaces then in use. The researches of M. Moissan have enabled him to classify the different simple bodies; the carbides, in fact, from being looked upon as ill-defined compounds, are now shown to possess new properties, so well defined that they serve as a good basis for the classification of the elements into natural groups. The property of decomposing cold water and furnishing absolutely pure acetylene gas is characteristic of the carbides of lime, barium, and strontium, while the carbides of aluminium and glaucinum—under similar conditions—will produce pure methane; these are two distinct and new reactions of great importance.

We will not, however, dwell longer on this subject—it has previously been published at some length in these columns, but will close this notice by expressing our admiration for the excellent work done and the great amount of time and labour M. Moissan has devoted to his research.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société d'Encouragement pour l'Industrie Nationale. Series 5, Vol. ii., No. 4.

Analysis of the Fourth Report of "The Research Committee on Alloys" of the Institution of Mechanical Engineers of London.—This is a long and interesting review of Prof. Roberts-Austen's report to the Institution of Mechanical Engineers and Engineering on February 12, 1897, but it is not suitable for abstraction.

Revue Generale des Sciences Pures et Appliquées.
Nos. 7 and 8.

Neither of these numbers contain any original matter of chemical interest.

Bulletin des Travaux de la Société de Pharmacie de Bordeaux. April, 1897.

Detection of Oil of Arachis in Olive Oil.—Prof. Ch. Blarez.—The detection of oil of sesame, cotton-seed oil, &c., in olive oil is not a matter of any great difficulty, but the problem of detecting the presence of oil of arachis is always a matter of delicacy. The author considers that M. Renard's method of detecting this adulterant has the fault of being too long and requiring great experience on the part of the person using it, and he proposes a new and much quicker method, based on the property its potash soaps possess, of being almost insoluble in strong, cold alcohol, in presence of a notable excess of potash. The originality claimed consists in the special procedure followed and the use of very simple apparatus, and may be briefly described as follows:—1. Pour 1 c.c. of the oil under examination into a test-tube 15 c.m. long. 2. Add 15 c.c. of pure 90° alcohol containing 4 or 5 per cent of pure potash. 3. Fit the tube with a vertical condenser. 4. Heat the tube carefully for about twenty minutes; the oil will rapidly saponify and disappear, the alcohol returning into the tube. 5. Remove the condenser, cork the tube and put it in a cool place. In the case of pure oil of arachis the contents of the tube became solid after the lapse of twenty-four hours. With pure olive oil there is no sign of solidification after the lapse of twenty-four, forty-eight, or even seventy-two hours; but with a mixture of the two there is always a flocculent precipitate, in which can be distinguished crystals of arachidate of potash.

MISCELLANEOUS.

Royal Institution.—The Friday Evening Discourse at the Royal Institution next week (May 28th) will be delivered by Prof. H. Moissan (Directeur Laboratoire de Chimie Minérale à l'Ecole Supérieure de Pharmacie, Membre de l'Académie des Sciences, Paris), who will lecture in French on "The Isolation of Fluorine" (with experiments). On Friday, June 4th, Mr. W. H. Preece, C.B., F.R.S., will lecture on "Signalling through Space without Wires." On Friday, June 11th, Mr. William Crookes, F.R.S., will deliver the last of the Friday Evening Discourses for the year; his subject will be "Diamonds."

The General Italian Exhibition.—This Exhibition will be held in Turin in 1898. A special section will be

devoted to Electricity, and a prize—called the "Galileo-Ferraris Prize"—will be given to the most important application of electricity to industrial purposes. The amount already subscribed towards this prize reaches 15,000 francs.

The Rose Polytechnic Institute of Terre Haute, Indiana.—The Calendar of this Institute now before us gives a complete list of the lectures and courses of instruction included in its curriculum. It is, firstly, an Engineering College; this naturally includes scientific training in such subjects as Chemistry, Electricity, Mathematics, &c. The general plan of instruction is based on the principle that laboratory and shop work, field and office practice, should all go hand-in-hand with theory and book work.

The Pennsylvania State College.—We have received the Prospectus and Syllabus of the above-named College, and have much pleasure in noting its growing importance and the extended scope of its teachings. Started in 1859 as the "Farmer's High School," for the purpose of giving a purely agricultural education, it has been so enlarged as to include all those subjects which lie at the foundation of modern industrial pursuits. The several courses of lectures, and of instruction generally, are grouped into seven Schools,—such as the School of Natural Science (biology and chemistry), the School of Mathematics and Physics, the Schools of Mines, Engineering, and so on. The course of chemistry lasts for two years, and includes both mineral and organic chemistry, theoretical and practical. Electricity is of course not neglected, with its many modern applications, while another peculiar feature of the College is its military organisation.

Disinfection with Formic Aldehyd.—In the Sanitary Chronicles of the parish of St. Marylebone, for the month ending March 31st, 1897, Dr. Winter Blyth records some interesting and valuable work done in connection with the disinfecting properties of formic aldehyd, commercially known when dissolved in water as "formalin" or "formol." The antiseptic powers of formic aldehyd are extraordinary; 1 part in 10,000 suffices to preserve milk, soup, and similar articles, for a considerable time, which fact naturally suggested that it might have true disinfecting powers. The aqueous solution called formalin does not give very satisfactory results when exposed in open dishes, as it has the peculiar property of changing into a white solid,—in other words, polymerisation takes place, CHOH changing into $\text{C}_2\text{H}_3\text{O}_3\text{H}_2$, the latter substance being a far less efficient disinfectant. Bouse and Boudet, however, found that when the gas is dissolved in a solution of calcium chloride, and afterwards heated under pressure, practically dry formic aldehyd was driven off. The ordinary method of disinfecting a room is to seal and plug up every crack and opening; then open a bottle of compressed sulphurous acid, and leave the room undisturbed for about twelve hours or more. A comparative trial was made with sulphurous acid and formic aldehyd, in two rooms in which were exposed, on bits of linen, four cultures of diphtheria, four of the typhoid bacillus, four of tuberculosis, and four of anthrax; there were also samples of coloured paper and materials placed in the room, to enable the officials to observe the effect, if any, on colours. After nineteen hours the rooms were opened, and the infected pieces of linen were sent to Prof. Macfadyen, who reported as follows:—

	Sulphur gas.	Formic aldehyd.
I. Diphtheria bacillus.	No growth.	No growth.
II. Typhoid bacillus.	Good growth.	No growth.
III. Anthrax bacillus.	Good growth.	No growth.

The tubercle samples could not be properly reported on, as they were found to be contaminated with other micro-organisms. The formic aldehyd room, on being entered, smelled strongly of the gas, but beyond making the eyes smart there was not much inconvenience for a short

while; but in the sulphur gas room three attempts, in as many quarters of an hour, were made to open the windows, and in each case Dr. Blyth was compelled to retire, gasping and choking; it was more than an hour before the windows could be opened. As for the colours, both gases showed a trivial bleaching in the case of silk when very carefully compared, but in the dyed papers no alteration whatever could be discerned. In conclusion, Dr. Blyth considers that formic aldehyd gas is superior to sulphurous acid gas as a disinfectant, and he recommends its adoption by the Vestry.

Sophistication of Foods.—We learn from the *Chem. Zeitung* that the Sanitary Department of San Francisco is publishing a black list of the manufacturers and dealers in adulterated food, with the names and addresses of the offenders in full.

Memorial to Professors Gauss and Weber.—We have the pleasure of calling the attention of our readers to the project for erecting, at Göttingen, a monument to the late Professors Gauss and Weber. A sum of 23,520 marks has been already collected for the purpose. Further subscriptions will still be welcomed, as the design of Prof. Harza (of Berlin) is of a somewhat expensive character. The work will be completed within two years. The President of the Committee is Prof. Dr. W. Vogt, and the Treasurer is Siegfried Bonfey.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Filling for Joints.—Can any of your correspondents recommend a good filling for open joints in a laboratory bench?—F. M.

MEETINGS FOR THE WEEK.

- MONDAY, 24th.—Society of Arts, 8. (Cantor Lectures). "Design in Lettering," by Lewis Foreman Day.
TUESDAY, 25th.—Royal Institution, 3. "The Heart and its Work," by Dr. Ernest H. Starling.
WEDNESDAY, 26th.—Society of Arts, 8. "Silver and Prices—the Economic Drain of Debtor Nations," by M. Frewen, B.A.
THURSDAY, 27th.—Royal Institution, 3. "Burke and the Revolution," by Churton Collins, M.A.
FRIDAY, 28th.—Royal Institution, 3. "The Isolation of Fluorine," by Prof. H. Moissan.
— Physical, 5. "The Perception of Phase Difference by the Two Ears," by Dr. A. A. Gray. "The Isothermals of Isopentane," by Mr. Rose-Innes.
SATURDAY, 29th.—Royal Institution, 3. "Music in England during the Reign of Queen Victoria," by J. A. Fuller Maitland, M.A.

IMPORTANT NOTICE.

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1957.

AN ATTEMPT TO CAUSE HELIUM OR ARGON TO PASS THROUGH RED-HOT PALLADIUM, PLATINUM, OR IRON.*

By WILLIAM RAMSAY, F.R.S., and MORRIS W. TRAVERS.

To chronicle experiments which produce no result is a necessity, although not entirely an agreeable one. Whatever the reason of the passage of hydrogen through red-hot iron, and through moderately heated palladium, and platinum—whether it be due to the solubility of the gas in the metal, or to the formation of an easily decomposable compound—neither argon nor helium is able to pass through any one of these metals, even at a fairly high temperature. This would imply their inability to form any compound, however unstable, with these metals, or to dissolve in them at a red heat. Such inactivity is in accordance with their general behaviour, and is still another proof of their inertness.

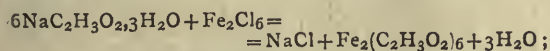
The experiment was made in the following manner:—A tube of hard, infusible glass was connected at one end with the reservoir of the gas under experiment, helium or argon. Into its other end was corked a tube of platinum, closed with a palladium cap, or, if iron was the metal under experiment, with a tube of thin wrought iron, also closed at the end; the closed end of the interior tube was placed so that it could be raised to a bright red heat by bringing a blowpipe flame to bear on the hard glass tube. The open end of the metal tube was cemented to a glass tube, attached to a Töpler pump, and provided with a Plücker's vacuum tube, so that the spectrum of any gas passing through the metal could be observed. This afforded, at the same time, a most delicate test of the presence of the gas under experiment. The metal tube was exhausted, until green phosphorescence appeared in the vacuum tube, and the gas, helium or argon, was admitted into the space between the glass and the metal tube, at atmospheric pressure. The glass tube was then heated to the highest temperature attainable with a blowpipe—perhaps 900° or 950° C. In no case, whether the metal tube consisted of palladium, platinum, or iron, was there the smallest transpiration of gas, even after half an hour. The phosphorescent vacuum remained in all experiments quite unimpaired.

SEPARATIONS WITH ALKALINE ACETATES. (PRELIMINARY PAPER).

By HARRY BREARLEY.

It has been previously shown (CHEMICAL NEWS, xxvii., 14; lxxv., 13) that the separation of iron from manganese by means of soda or ammonia acetates is the more imperfect the larger the excess of acetate used. It has also been shown (CHEMICAL NEWS, xl., 273; lxxiv., 16) that for this and other metals the effect of excessive acetate was obviated by the presence of free acetic acid.

The reaction between ferric chloride and soda acetate has been written—



whence it follows that 8 grms. of the crystallised soda acetate ($\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$) would be needed to precipitate 1 grm. of metallic iron existing as ferric chloride. But iron solutions are never precipitated in that state. Pure or carbonated alkali is added; after the free acid is neutralised, the precipitated hydrate continues to be dissolved by the ferric chloride to saturation point. When this is reached, the faint excess, taken up in two or three drops of HCl, marks the point, commonly called "neutralised," to which solutions are usually taken before adding the acetate. Acetate is needed only to precipitate that portion of the ferric chloride not already precipitated as hydrate, and which holds the precipitated hydrate in solution. The amount theoretically required will bear the same ratio to 8 grms. that the final bears to the initial quantity of Fe_2Cl_6 when 1 grm. of metal is operated on. It may be of interest to show experimentally what amount of acetate is necessary to precipitate a solution containing variable quantities of ferric chloride and hydrate. It is certain that many an imperfect separation may be redeemed by using minimum amounts of acetate; and separations, generally considered impossible, effected by further modification in the same direction.

It was decided to answer questions similar to the ones suggested when the iron solutions contained—(a) No free acid and no dissolved hydrate; (b) half the total soluble hydrate; and, (c), total soluble hydrate, i.e., as much hydrate as the solution could be made to dissolve. More or less completely, the same series was gone through with varying amounts of acetic acid and with soda and ammonia salts.

The iron solution chiefly used was made by dissolving 40 grms. of Swedish bar iron in 290 c.c. HCl and oxidising with 50 c.c. HNO_3 . There was prepared also a solution of iron in HNO_3 (1·20), and a solution by dissolving in HCl, oxidising with HNO_3 , evaporating to dryness, and re-dissolving in HCl. The two latter solutions were intended for comparing with the first at different points, so that the data established might be available whichever method should be used for preparing the iron solution. All three are recommended in one or other of the modern text-books.

The amount of free acid was determined by adding excess of normal soda carbonate to boiling iron solutions, filtering off aliquot part, and determining excess of alkali with standard acid and methyl-orange (Sutton, "Vol. Anal.," 5th ed., p. 89).

Dissolved Hydrate.

For determining the dissolved hydrate, iron solution representing $\frac{1}{2}$ grm. of metal was placed in a flask and normal soda or ammonia carbonate run in from a burette. Towards the end the alkali was added very slowly, and plenty of time allowed for the precipitated hydrate to dissolve. The faint permanent precipitate was dissolved with standard HCl. The total alkali used, less that needed for neutralising the free acid, gives the quantity

TABLE I.
With Soda Carbonate.

Iron solution.	Total alkali. C.c.	To precipitate			Ratio, total to final Fe_2Cl_6 .
		Required by free acid.	Dissolved hydrate.	Total hydrate. C.c.	
Aqua regia..	37·74	14·2	23·54	26·8	8·35
	37·84	14·2	23·64	26·8	
Hydrochloric acid..	33·72	10·35	23·37	26·8	7·93
	33·82	10·35	23·47	26·8	
Nitric acid..	34·0	10·1	24·0	26·8	9·57
	34·0	10·1	24·0	26·8	

With Ammonia Carbonate.

Aqua regia..	37·84	14·2	23·64	26·8	8·48
Hydrochloric	33·93	10·35	23·58	26·8	8·32

* A Paper read before the Royal Society, May 14, 1897.

TABLE II.

	Free acetic	Soda acetate.				Ammonia acetate.		
		0	5	10	20 c.c.	0	5	10 c.c.
N. H.	{ Acetate	145 c.c.	150	160	175	150	155	163
	{ Temp. turbidity ..	84° C.	90	94	95	86	94	90
H. H.	{ Acetate	73	75	80	90	78	80	85
	{ Temp. turbidity ..	74	80	82	80	80	91	92
T. H.	{ Acetate	5*	6	12	16	5*	7	10
	{ Temp. turbidity ..	?	80-84	80-84	88	?	80	81

needed to form the dissolved hydrate. These solutions, on standing, become gradually turbid, and after standing weeks or months allow the precipitate to settle somewhat, but such a precipitate cannot be separated by ordinary filtration. Completely "neutralised" and perfectly clear solutions will appear slightly turbid by reflected light after standing over night. This additional precaution was taken in determining the dissolved hydrate. Some results are arranged in Table I.

These solutions all contained alkaline salts, due to the neutralisation of the free acid. On this account they do undoubtedly require less alkali. The nitrate is less active in this respect than the chloride, and this may partly explain why more hydrate is dissolved in the nitric than the aqua regia solution, and least of all in the HCl solution. When ammonium chloride representing 10 c.c. strong HCl was added to a solution the alkali required to precipitate the dissolved hydrate fell from 23.57 c.c. to 22.74, and with nitrate representing 10 c.c. HNO₃ (1.42) it fell to 22.87 c.c.

Kessler (CHEM. NEWS, xxvii., 14) states:—"When a hydrochloric acid solution of perchloride of iron is neutralised by means of soda carbonate, so as to cause a permanent precipitate, and the latter is cautiously dissolved by the addition of some HCl, a liquid is obtained which contains fourteen times its equivalent of ferric hydrate in solution, yet it is not precipitated by boiling." This passage seems to have been misread by Blair ("Chem. Anal. of Iron," p. 108), who makes out that, *theoretically*, half a gram. of soda acetate is sufficient to precipitate 1 gram. of iron, as though fourteen times as much iron exists as dissolved hydrate as exists as chloride. The intended meaning seems to be that the dissolved Fe₂(HO)₆ = 14 Fe₂ (existing as Fe₂Cl₆). Read this way, the two results are broadly confirmatory.

Precipitating the Iron.

The precipitations with acetate were performed in flasks. One gram. of iron taken in all cases, and made, finally, in the cold, up to 480 c.c., so that at boiling-point it would measure 500 c.c. approximately. The temperature at which the heated solution became turbid was observed. In some cases after a faint turbidity the formation of the precipitate was very tardy, and is represented by two observations: *e.g.*, 76-80° C., 20 c.c. of the soda acetate contains 1 gram. Na₂C₂H₃O₂·3H₂O crystals. The ammonia acetate was made by neutralising acetic acid (33 per cent) with ammonia. One and a half c.c. of such a solution contained as much acetic acid as 1 gram. soda acetate crystals, so that they were made of equal value by making 1.5 c.c. of the former up to 20 c.c. The solutions were neutral on the acid side, if I may be allowed to use such contradictory terms.†

An iron solution may be said to be completely precipitated when, the basic acetate having settled, the supernatant solution is colourless and free from iron. This, agreeable to theory, was found to be the case when-

having no free acid and no dissolved hydrate, 8 grms. of soda acetate crystals were added; 7½ grms. left a faintly coloured supernatant solution. If this faintly coloured solution is passed hot through an asbestos filter, the filtrate is colourless, crystal clear, and when acidified gives no colouration with sulphocyanide. A similar result is obtainable using 7½ grms. It is this property of an asbestos filter which it is more particularly desirable to observe; for is it not likely that, if only so much acetate is added as will leave a faintly coloured supernatant solution, an associated metal, having similar properties, may remain completely in solution and depend for its complete separation from the last portion of iron on its deportment to the asbestos? A very considerable quantity of iron may be removed from a hot solution in this way; cold solutions pass unaltered.

No great labour was spent, therefore, in determining how much acetate was needed to give a colourless supersolution after the settling of the precipitated acetate, but rather to determining with how little acetate an iron-free filtrate could be obtained. There is, of course, no absolutely unalterable amount. Something depends on how the filter is made, how long the solution is boiled, what amount of alkaline salts, &c. In the instances tabulated the solution was heated until it quite boiled, cheesed until the precipitate settled, and filtered. Where soda acetate is used for precipitating, soda salts have been used throughout, and similarly for ammonia. N.H., H.H., and T.H. mean that the iron solution contained no hydrate, half the total hydrate, and the total hydrate respectively. The table shows the minimum acetate.

The solutions marked with an asterisk became turbid on adding the acetate, but did not leave a clear supersolution unless they were heated. When larger amounts of acetate (10 c.c.) were added, the precipitate settled, without heating, leaving a perfectly colourless supersolution.

The preparation of the large vols. of T.H. solution enabled me to confirm the figures given above for the amount of hydrate soluble in ferric chloride.

A dozen or so precipitations were made of iron solutions to which ammonia salts, containing respectively 10 c.c. HCl and 10 c.c. HNO₃, had been added. Their presence favours the precipitation, the chloride more decidedly so than the nitrate. With the same volume of acetate the temperature of turbidity falls about 10° C.

Precipitating from more dilute solutions did not seem to make much difference. The tendency was to give more perfect separations, but this may be due to the longer digestion of the larger volume between precipitating and boiling points.

The present object is to find with what amounts of acetate, dissolved hydrate, and free acetic acid, the most effective separations may be made of those metals which are at all separable by these means. Attention is specially to be paid to those separations which are of value to the steel works' chemist, and hence separations from iron claim first notice. Qualitative tests, which have indicated at least the partial separation of ferric oxide and alumina, for instance, lend colour to the hope that the completed investigation may not be without value.

The Laboratory, Norfolk Works, Sheffield.

* The neutralised solutions I obtained, if diluted with about 200 c.c. of water, were partly precipitated by boiling.

† The ammonia acetate was made two months before using. It was then neutral, as when first made. It has been stated that the solution decomposes on keeping, and should be made as required. I believe this is not a general practice, and this point is specially noted in the hope that it may elicit further information.

CONTRIBUTION TO THE STUDY OF
HYPONITROUS ACID.*

By A. HAUZSCH and A. L. KAUFMANN.

(Continued from p. 245).

Hyponitrite of Benzyl, $C_7H_7ON = NOC_7H_7$.

THE only ether of hyponitrous acid yet known, hyponitrite of ethyl, was obtained by Zorn in the form of a yellow oil, by treating iodide of ethyl with hyponitrite of silver. In spite of the explosibility of this ether, its vapour density has been determined. It corresponds to the formula $(C_2H_5)_2N_2O_2$. The benzylic ether which we have prepared is much more stable.

Pure, dry, hyponitrite of silver is added in excess, to a well-cooled etherised solution of carefully purified iodide of benzyl; the temperature, which will tend to rise, must be kept low. When, after two or three hours, the smell of iodide of benzyl has disappeared, it is filtered, the ether is evaporated off, and we obtain the benzylic ether in a pure state by re-crystallisation in ligroin.

- I. 0.1883 grm. of the substance gave 0.4757 grm. CO_2 and 0.1042 grm. H_2O .
- II. 0.1147 grm. of the substance gave 0.2905 grm. CO_2 and 0.0639 grm. H_2O .
- III. 0.1460 grm. of the substance gave 14.5 c.c. N at $17^\circ C$. and 760 m.m. pressure.

	I.	II.	III.	Theory, C_7H_7NO .
C.	68.83%	69.07%	—	69.42%
H.	6.15%	6.19%	—	5.79%
N.	—	—	11.53%	11.57%

The molecular weight of the ether has been determined cryoscopically in acetic solution, by passing—as recommended by Beckmann—a continuous current of dry air over the liquid.

- I. 0.1041 grm. of the substance, dissolved in 20 c.c. of glacial acetic acid (sp. gr. = 1.055), lowered the congealing point by 0.081° (mean of six observations).
- II. 0.3086 grm. of the substance, dissolved in 20 c.c. of glacial acetic acid, lowered the congealing point by 0.251° (mean of seven observations).

Molecular Weight.

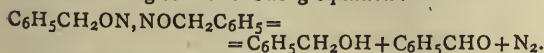
	Found.	Theory, $C_{14}H_{14}N_2O_2$.
I.	236	242
II.	227	

Hyponitrite of benzyl is bi-molecular, as is free hyponitrous acid and Zorn's ethylic ether. It is easily soluble in alcohol and ether, but only slightly so in ligroin. The latter precipitates it from its solution in alcohol or ether. It melts at 43° to 45° with decomposition, and detonates when rapidly heated to 60° . Even at the ordinary temperature it volatilises easily. In an experiment made, its loss of weight was 7.5 per cent in seventeen hours.

The decomposition of hyponitrite of benzyl when warmed gives rise to a considerable disengagement of gas. To discover the nature of this gas we dissolved a weighed quantity of the ether, in methylic alcohol, warmed the solution to 50° in a current of carbonic acid, and collected the gas in a nitrometer.

0.1915 grm. gave 17.7 c.c. (at 18° and 751 m.m. pressure) of nitrogen (and not protoxide of nitrogen).

Nitrogen found 10.57 per cent; theory 11.57 per cent. Assuming that the nitrogen is liberated nearly quantitatively, the decomposition of hyponitrite of benzyl is no doubt according to the following equation:—



This experiment confirms Zorn's observation relative to the decomposition of the ethylic ether of hyponitrous acid.

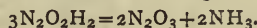
Decomposition of Hyponitrous Acid.

It has been generally admitted, up to the present, that hyponitrous acid splits up quantitatively into protoxide of nitrogen and water.

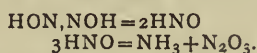
The fact that hyponitrous acid gives a blue colour to iodide of potassium and starch acidulated with acetic acid, then the brown colouration produced by the addition of ferrous sulphate and the blue colouration produced by the addition of a phenylamine to a sulphuric solution of hyponitrous acid, and, again, the observations made on the determination of the conductivity of this acid, which will be described further on,—all these facts go to prove that, by a secondary reaction, hyponitrous acid gives birth to nitrous acid.

Above all we could suppose that, by the inverse of the synthesis of hyponitrous acid achieved by Wislicenus, this acid would split up, under certain conditions, into nitrous acid, and hydroxylamine according to the equation $HON, NOH + H_2O = HONO + NH_2OH$.

In spite of all our efforts we have not been able to prove the presence of hydroxylamine among the products of decomposition of hyponitrous acid. On the other hand, we have shown that hyponitrous acid partially splits up into nitrous acid and ammonia, according to the equation—



This decomposition is easily explained on the supposition that the acid, HON, NOH , becomes first changed into its tautomeric demi-molecular form, O, NH , to which nitroso-benzene corresponds as its ether; after which a molecule of this aldehyd of nitrous acid becomes oxidised, after the manner of true aldehyds, at the expense of another molecule, which is thus reduced to ammonia:—



As a secondary reaction, the molecule of ammonia may be oxidised at the expense of the half molecule of nitrous anhydride formed, and may eventually form nitrogen, water, and nitrous or nitric acid, according to the equation $NH_3 + N_2O_3 = N_3 + H_2O + HNO_3$. However, in comparison with the splitting up into protoxide of nitrogen and water, the decomposition we have just mentioned takes place but very slowly in aqueous solution. A freshly prepared solution of hyponitrous acid, boiled in a flask fitted with a vertical condenser, became completely neutral, and did not contain a trace of nitrous acid. But, on the contrary, an exactly similar solution left for several hours at 25° before being boiled, maintained its acid reaction to the end of the operation, and, moreover, contained 2 per cent of free acid. When neutralised and evaporated to dryness, it showed the nitrite reactions very clearly.

Another portion of the same solution was similarly boiled, and then evaporated to dryness with hydrochloric acid. Ammonia could be detected in the residue by means of Nessler's solution.

In another experiment a freshly prepared solution of hyponitrous acid was divided into three portions; the first portion was left intact, the second was treated with caustic potash in excess, while to the third a few drops of hydrochloric acid were added. After the lapse of twenty-four hours, these three solutions were analysed. In the first, which had been hyponitrous acid alone, we proved the presence of nitrous acid by means of diphenylamine. The complete absence of ammonia, and the presence of unaltered hyponitrous acid. The second contained neither nitrous acid nor ammonia, but there was still some hyponitrous acid present. The third contained neither nitrous acid, ammonia, nor hyponitrous acid, but nitric acid. These three experiments confirm the view that hyponitrous acid is most stable in an alkaline solution, and least stable in

* *Moniteur Scientifique*, vol. xi., p. 336, May, 1897.

an acid solution. The stability of the pure acid in aqueous solution is midway between the two.

We need not be astonished that no ammonia is formed in these last three experiments. The ammonia which might be formed in very small quantities in the acidulated solution, would be immediately destroyed by the action of the nitrous acid which would be formed simultaneously. We can only detect the presence of ammonia by the very rapid evaporation of strongly acid solutions of hyponitrous acid.

The formation of nitrous acid during the decomposition of hyponitrous acid explains the blue colouration obtained with diphenylamine, and the brown colouration with ferrous sulphate. This reaction equally explains the fact that the determination of the rapidity of the decomposition of hyponitrous acid into protoxide of nitrogen and water has not been found possible.

Before recognising this secondary reaction we quite expected that the relation between the still undecomposed acid (a) and that already decomposed into protoxide of nitrogen and water (x) could be determined at any or every moment by the diminution of the acid titration. By the equation—

$$c = \frac{x}{t} \cdot \log \frac{a}{a-x}$$

we could have measured the speed-constant of the decomposition. With this idea we took, at stated intervals, 10 c.c. samples of a solution of hyponitrous acid, prepared at 0° and kept at 25° in an Ostwald thermostat. These samples were titrated with a 1/16th normal solution of baryta, using phenolphthalein as indicator. But the values of c , calculated according to the above formula, were not even approximately constant, and moreover increased as the experiment went on. This increase in the speed of decomposition can only be attributed to the gradual acceleration of the secondary reaction, which results in the formation of nitrous acid. It is another example of accelerant catalytic action of the ions of hydrogen, for here again the presence of nitrous acid has been definitely proved.

The fact that acid considerably accelerates the decomposition of hyponitrous acid has been shown in a special experiment, by determining the speed of decomposition in the presence of hydrochloric acid. The initial values of c were in this case much greater than those obtained with an aqueous solution of pure hyponitrous acid. But here again we could not obtain a constant by the diminution of the titration of the acid. But if we took as abscissæ the calculated values of

$$e = \frac{x}{t} \cdot \log \frac{a}{a-x},$$

and the corresponding intervals of time as ordinates, we obtained a regular curve which showed that the speed of decomposition increased with the quantity of the substance decomposed. We propose later to make a special study of this phenomenon.

(To be continued).

A NEW AND ACCURATE METHOD FOR THE ESTIMATION OF POTASSIUM.

By H. N. WARREN, Principal, Liverpool Research Laboratory.

THE solution containing the alkalis as chlorides, having been previously exhausted of the accompanying group metals, is heated with an excess of platinic chloride, and the whole evaporated to very small bulk in a platinum dish, or other suitable receptacle; to the contents are now added about double the original quantity of a mixture composed of equal parts of amylic alcohol and ether. The precipitate is by these means immediately

rendered dense, and can thus be washed once or twice with the utmost facility, using the same mixture. The yellow precipitate thus obtained is next transferred to a small glass beaker, and heated to the boiling point with the addition of about 5 c.c. of formic acid. The solution thus speedily assumes a brownish tint, at which stage a slight excess of ammonia is introduced, and re-boiled, when the whole of the platinum is precipitated in the form of black flocks, which may be readily washed and dried, from the weight of which the percentage of potassium present may be readily calculated.

With a little practice the operation will be found more expeditious, more accurate, and at the same time less troublesome, than the general methods advised for the estimation of potassium.

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ON THE RECOGNITION OF THE YELLOW OF NAPHTHOL S, AND OF ANALOGOUS COLOURING-MATTERS IN WHITE WINES AND LIQUEURS.

By ALBERTO D'AGUIAR and WENCESLAU DA SILVA.

THE yellow of naphthol S, like diamond brilliant yellow S, &c., are scarcely extracted from an alkaline solution by means of solvents, such as amylic ether, acetic ether, sulphuric ether. Under these conditions we proceed as follows:—

A portion of the wine is strongly acidified with sulphuric acid, and shaken up with amylic ether, which extracts all the colouring-matter derived from the coal-tar and a part of the natural colouring-matter of the wine. After decantation and filtration the amylic alcohol is agitated with an excess of ammonia, and allowed to remain at rest until it is permanently limpid. The natural colouring-matter of the wine, as also various other matters, are precipitated by the ammonia. The amylic alcohol retains in solution a portion of the coal-tar colour sufficient for its detection by dyeing-tests or by reagents. The amylic solution is shaken up with water and acidulated sulphuric acid; after standing it is evaporated, in contact with a thread of silk, with a few drops of ammonia. The silk is then distinctly dyed, and the residue left by the amylic alcohol is next submitted to the action of sulphuric and hydrochloric acids and ammonia, to allow of a study of the changes produced by these reagents.

We have made these experiments on wines coloured with naphtha yellow S, brilliant yellow S, diamond yellow, turmeric and fustic, as well as on natural wine. The results were positive with the first three, but negative with the last three, notwithstanding the strong colouration of the amylic-alcoholic tinctures.

Bellier's procedure also gave very distinct positive results with the first three tests, but negative with the latter.

These experiments have been repeated on Ermida wine mixed with the other yellow coal-tar colours mentioned in a former paper. The results have been also positive.—*Comptes Rendus*, cxxiv., No. 18, 1897.

Contribution to the Study of Tinctorial Reactions.

—A. Reyckler.—After describing a number of experiments and verifications of the conditions existing, the author claims that he has not disproved the theory put forward by M. Knecht; but, on the contrary, he has confirmed the ideas held on the constitution and action of saline solutions in respect of dyeing.—*Bulletin de la Société Chimique de Paris*.

SOME PRESENT POSSIBILITIES IN THE ANALYSIS OF IRON AND STEEL.*

By C. B. DUDLEY.

To the analytical chemist there are few substances in nature more interesting than a piece of pig-iron, few substances which have received more study, and few which present chemical problems more difficult of solution. The amount of work which has already been done in connection with this very common but very complex substance is something enormous. Indeed, if we add to the study which has already been put on pig-iron itself, the work which has been done on what may perhaps fairly be called its progenitors, viz., the ores, the fuel, the flux, and the refractory materials used in its production, and then consider still farther the labour already expended in the analysis of what we may call the progeny of pig-iron, viz., castings, wrought iron, malleable iron, and the numerous grades and kinds of steel made by the various processes of the present day, we shall surely be safe in saying that more chemical work has been done in connection with pig-iron than with any other substance in nature. Is it too much to affirm that at the present time one-third, possibly one-half, of all the chemical work done in the world is in connection with the iron industry, either in the solution of unworked-out problems, the development of new methods of analysis, or in the routine analyses affecting the interests of producer and consumer.

But the amount of work already done and in daily progress in connection with this substance is not all that may be said in regard to it. The complexity of pig-iron is very great, and consequently the analytical problems presented are far from being easy of solution. It may not be uninteresting to enumerate some of the substances which have already been found in pig-iron. We find, besides the element iron, carbon, phosphorus, silicon, sulphur, manganese, copper, chromium, tungsten, titanium, vanadium, nickel, cobalt, aluminum, potassium, sodium, magnesium, calcium, and lithium. It is fair to say there is apparently well grounded belief that the last five are characteristic of intermingled slag rather than of the metal itself. It is not intended that it should be understood that all of these substances have been found in any one sample of pig-iron, but that all these substances have actually been detected in the analysis of this alloy. Indeed there seems no reason why any element, which either occurs in the metallic condition in nature, or which is reducible to that condition by carbon, and which is not volatile at the temperature of the blast furnace, may not occur in pig-iron, provided of course it will alloy with the metal. Quite a large number of other substances besides those mentioned above have actually been alloyed with some form of iron or steel. Among these may be mentioned zinc, tin, lead, antimony, bismuth, molybdenum, silver, platinum, rhodium, iridium, palladium, and gold. Nor is this all that may confront the analyst who devotes himself to the chemistry of iron and steel. Not less than three elements which usually exist in nature in the gaseous form occur in these metals, and are believed to have important influences on their physical properties. These are oxygen, hydrogen, and nitrogen; while the numerous analyses show the presence of carbon monoxide in both cast-iron, wrought iron, and steel. It seems quite evident that the chemist who hopes to successfully cope with the problems which are involved in even the ultimate analysis of iron and steel in their various forms, must be well equipped with a liberal share of the methods and processes known to mineral chemistry; and, on the other hand, if he attempt the proximate analysis of these substances, or the separation and determination of the various compounds of the elements present, with iron or with each other, he will at least be

brought on the border-ground of organic chemistry. Some of the carbon compounds which are characteristic of the brilliant work of the present President of the French Chemical Society, are known to occur in or have already been isolated from pig-iron.

It would lead us too far from our present purpose to do anything more than enumerate the largest number of the elements given above. Suffice it to say that in what follows, we shall confine ourselves to the five first mentioned, viz., carbon, phosphorus, silicon, sulphur, and manganese. And the question which we shall ask ourselves is, what is the present condition of a portion of the analytical methods for the determination of these substances, considering these methods both in regard to their accuracy and speed? One word of precaution. It would be manifestly impossible to comment on all the methods in use for determining these constituents. To enumerate them alone would weary your patience. We shall confine ourselves, therefore, principally to methods which may be, or are, used when the diverse interests of producer and consumer are involved.

Beginning, then, with total carbon in pig-iron, wrought iron, and steel, we deem it safe to say that the method by combustion in oxygen gas, as at present known and worked in many laboratories, leaves very little to be desired, so far as accuracy is concerned, and is sufficiently rapid for most commercial uses. The modification introduced some years ago, of using a solution of the double chloride of copper and ammonium, instead of simple chloride of copper,* to release the carbon from the iron, took away from the combustion method one of its greatest difficulties, viz., the long time required to dissolve the metal. This modification, as many will doubtless remember, reduced the time required for solution from two or three days to an hour or less. Indeed, at the present time, if a good stirring machine is used, it is quite possible to dissolve 3 grms. of fairly fine borings of pig-iron, wrought iron, or steel, in 200 c.c. of the proper solvent in from ten to forty minutes. Still further, the studies of the Committee on International Standards for the Analysis of Iron and Steel have further modified the method, and it is believed rendered it much more accurate. Among these modifications may be mentioned the use of an acid instead of a neutral or basic solution of the double salt to dissolve the metal. This point was thoroughly worked out by Blair (*Trans. Am. Inst. Mining Eng.*, xix., 614). Following this came the work done in the laboratory of the Pennsylvania Railroad Company, demonstrating the unreliability of the use of the double chloride of copper and ammonium as a solvent, owing, as appeared later, to the probable presence in all ammonia and its salts obtainable in the market, even those marked "C. P.," of some carbonaceous material, possibly pyridine (*Trans. Am. Inst. Mining Eng.*, xx., 242) derived from the gas liquor used in making the ammonia. The substitution of the potassium for the ammonium salt has apparently completely overcome this difficulty, and this, with the use of oxygen gas instead of lead chromate, in which to burn the carbon, and some modifications of the absorbing and purifying train (*Journ. Amer. Chem. Soc.*, xv., 448), have seemingly placed the dry combustion method for determining carbon in the front rank of successful and accurate analytical processes. The principal known source of error in the method at the present time appears to be in connection with the weighing. The potash bulbs and small calcium chloride tube used in

* It is difficult to say positively who first suggested this modification. The first mention in literature that we are able to find is in the *Transactions of the American Institute of Mining Engineers*, iv., 157, by J. B. Pearse. But a private communication from Andrew S. McCreath, states that he made the suggestion while working under Pearse, and that Professor Richter, in the "Leoben Jahrbuch," had previously suggested the use of potassium or sodium chloride with copper chloride, which led him to try the ammonium salt. McCreath's description of the method, as used by himself, is published in the *Transactions of the American Institute of Mining Engineers*, v., 575.

* Presidential Address delivered at the Troy Meeting of the American Chemical Society, December 29, 1896. From the *Journal of the American Chemical Society*, xix., No. 2.

absorbing the carbon dioxide weigh altogether some 50 to 60 grms., and present considerable surface. If now, between the weighing before the combustion and the weighing after the combustion, the interval being an hour or a little more, there is considerable change in the hygroscopic condition of the atmosphere, an error of 0.01 per cent may be easily introduced. If we may trust our experience, it is difficult to make closely agreeing duplicate combustions in showery weather. Blair suggests a method of overcoming this difficulty, consisting in having a second potash bulb and calcium chloride tube of—almost as nearly as possible—the same size on the opposite end of the balance when weighing.

In regard to the accuracy of the method as at present understood, it may be said that undoubtedly the best test of the accuracy of a method is the recovery of a known amount of any substance added to the material to be analysed. This procedure being manifestly impossible in the case of iron and steel, we are compelled to judge of the accuracy of the combustion method, as applied to these metals, in some other way. For this purpose, however, we have at hand the results obtained by different chemists, using different methods, but working on the same samples. In the course of the work done by the Committee on International Standards for the Analysis of Iron and Steel, the carbon in four samples of steel was determined. First, by using acid double chloride of copper and potassium as solvent, and burning in oxygen gas; Second, by using the same solvent, and burning in chromic acid solution; and Third, by treating the borings direct with bisulphate of potash and heat, conducting the carbon monoxide and sulphur dioxide formed over hot solid chromic acid, which oxidised both gases and retained the sulphur trioxide formed, and finally measuring the volumes of the resulting carbon dioxide in an eudiometer tube. Each method was used by a different chemist. The results obtained are as follows, the letters at the side representing the four samples of steel, the figures at the top representing the chemists, and the figures in the columns the percentages of carbon in the steel samples:—

	1.	2.	3.
A	1.455(a)	1.440(a)	1.450(b)
B	0.815	0.800	0.815
C	0.450	0.450	0.448
D	0.152	0.185	0.168

(a) *Proc. Eng. of Western Penna.*, ix., [9], 35.

(b) *Ztschr. anorg. Chem.*, iv., [3] und [4], 505.

The agreement of the results on the first three samples is quite marked. The discrepancy on the fourth sample has not been explained. The matter is discussed in considerable detail in reference (a), but we think it safe to conclude that, so far as method goes, the determination of total carbon in pig- or cast-iron, wrought iron, and steel, is reasonably accurate.

The speed of the combustion method as at present worked in good laboratories is quite remarkable, compared with the possibilities twenty-five years ago. A sufficient supply of sample borings being at hand, one operator using two furnaces may readily make from fourteen to sixteen combustions in a day of eight hours; it being understood that the bulbs are weighed with oxygen gas in them instead of air, and that the last weight of each combustion, except the last one at night, is taken as the first weight of the succeeding one. It is, of course, assumed that when turning out the amount of work above described, the furnaces and apparatus are all in good order, and everything working well. Accidents, an occasional overhauling of the apparatus, blank combustions from time to time for testing purposes, and once in a while an obstinate steel that refuses to dissolve in time or gives trouble in filtration, will tend to diminish output. The results obtained with this rapid work show, when duplicates are made, occasional discrepancies, as high as three hundredths of a per cent in a steel containing 1 per

cent of carbon, but we have seen very large numbers of duplicates, made as above described which did not disagree one one-hundredth.

Again, when work is not so plentiful as to admit of the procedure described above, the method still permits satisfactory speed. Starting with a fresh sample of borings and everything in good order, but cold, it is not difficult to get two closely-agreeing determinations on the same sample in two hours and a half. Of course, in investigation or referee work, more time would undoubtedly be used, especially if the interests involved are very great. But we have many times been astonished in our own laboratory at the close agreement between the results obtained in the rapid manner described above, and the duplicate analysis made on the same sample for confirmatory purposes, but using much more time and pains.

Turning now to the determination of combined carbon and graphite, we do not find the state of affairs so satisfactory. As is well known, these two constituents are usually found by first determining total carbon, then dissolving another portion of the sample in hydrochloric acid, filtering, and washing with caustic potash, alcohol, and ether, and then burning the residue; collecting and weighing the carbon dioxide formed, as in an ordinary combustion. The result is called graphite, and the combined carbon is the difference between the total carbon and the graphite. But, as Shimer (*Trans. Am. Inst. Min. Eng.*, xxv., 395) has so well shown, what we actually get by this procedure is not necessarily the graphite and the total combined carbon in the sample, but only the combined carbon which exists in the metal as a carbide soluble in hydrochloric acid. If the sample contains carbides not soluble in that acid, nor in the materials used in washing, the carbon of these carbides appears with, and is counted as, graphite. Shimer shows that titanium, and possibly vanadium carbide, are apparently not infrequently thus counted. The use of sulphuric instead of hydrochloric acid leads to the same error, while the employment of nitric acid as solvent, apparently gives the graphite much more definitely, but leaves us in doubt as to whether the combined carbon is really the combined carbon which we want, in order to have light on the quality of the metal we are dealing with. It is obvious that the difficulty here is in our lack of knowledge as to what carbides actually exist in pig- and cast-iron, and if there are several of them, which one or ones do we actually want to know the carbon content of. If we knew positively that the combined carbon wanted was that which exists in the metal as carbides of iron and manganese, and that these carbides were soluble in hydrochloric or sulphuric acid, while all other carbides present were not soluble in these acids, obviously we should use these acids when determining combined carbon. On the other hand, if we want to know only graphite, and care little about the combined carbon, apparently nitric acid is the solvent to use. It is clear that much more work is needed on this subject—a state of affairs which, as we progress, we shall find is characteristic of other constituents of the metals we are considering.

(To be continued).

Researches on the Composition of Wheats and on their Analysis.—Aimé Girard.—The exclusion from human consumption of 30/100 of the mass of wheat known as "low produce" and refuse milling, places at the disposal of agriculture a very rich residue for the food of cattle. These products are very rich in fatty matter, but their woody nature presents a certain resistance to the action of solvents. To facilitate this action, I advised, some years ago, to moisten these products with hydrochloric acid at 5 per cent, and to dry them afterwards, so as to transform the celluloses and the vessels into pulverulent hydrocellulose, which is easily penetrated by benzene or ether.—*Comptes Rendus*, cxxiv., No. 18.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, April 29th, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

MESSRS. Arthur Croft Hill, Edward G. Guest, and Horatio Ballantyne were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Gerald Noel Brown, 8, The Esplanade, Plymouth; George Lawson Johnston, Kingswood, Sydenham Hill, S.E.; William Taverner, 1, Stapenhill Road, Burton-on-Trent.

The PRESIDENT announced that the Council had ordered a re-count of the balloting papers handed in at the Anniversary Meeting. The re-count would be conducted by the two Scrutators appointed by the Society at the Anniversary Meeting, in the presence of the Secretaries.

Mr. CASSAL enquired whether the actual numbers would be announced.

The PRESIDENT replied that he assumed that they would.

Of the following papers those marked * were read:—

*53. "On the Explosion of Chlorine Peroxide with Carbonic Oxide." By H. B. DIXON, M.A., F.R.S., and E. J. RUSSELL, B.Sc.

To test the question whether dried carbonic oxide is more readily attacked by "nascent" than by ordinary oxygen, the authors have fired a dried mixture of chlorine peroxide and carbonic oxide. They find that the carbonic oxide is not completely burnt in the flame although the oxygen is in excess; and the drier the mixture the less carbonic oxide appears to be burnt. The results do not favour the view that "nascent" oxygen attacks carbonic oxide more readily than ordinary oxygen.

DISCUSSION.

Dr. SCOTT thought it desirable to try the effect of some compound, such as chloride monoxide, which contained only one atom of oxygen, before concluding that "nascent" oxygen was inoperative.

Professor ARMSTRONG referred to the difficulty of ascertaining whether a gas was completely free from water.

The PRESIDENT thought that the ultra-violet spectrum of water vapour, which he and Professor Liveing and Dr. Huggins had simultaneously discovered, would be found to be a very delicate test of its presence in flames or explosive mixtures.

Mr. CROOKES agreed with the President as to the delicacy of this test.

*54. "On the Decomposition of Iron Pyrites." By W. A. CALDECOTT, B.A.

In "Watts' Dictionary" (1892 edition, vol. iii., p. 64) it is stated that ferrous sulphide "is formed by the reduction of Fe_2O_3 on ferric salts by decomposing organic matter in the presence of sulphates," also that "finely-divided yellow pyrites (FeS_2) oxidises in air forming chiefly FeSO_4 ." T. K. ROSE ("The Metallurgy of Gold," 1896, page 343) states that " FeS_2 is oxidised by air and water, FeSO_4 and free H_2SO_4 being formed."

In the treatment of auriferous pyritic Witwatersrand conglomerate, a large percentage of the ore is reduced by wet crushing in the battery to an impalpable powder. This fine material constitutes "slimes," and is carried in suspension by water into extensive dams, where it settles. The slimes leaving the battery are free from ferrous sulphide, but this compound may be detected in them a few days after deposition in the dams. The settled slimes form a clayey mass, practically impermeable to air and water, and consequently subsequent oxidation of the ferrous sulphate proceeds extremely slowly.

When iron pyrites are crushed to an impalpable powder in an iron mortar ferrous sulphide is formed.

It thus appears that ferrous sulphide and not ferrous sulphate may be the first product of the decomposition of iron pyrites.

Owing to the almost total absence of acidity in slimes deposited as above, even when containing 0.89 per cent ferrous sulphide, it is probable that under these conditions FeS_2 undergoes dissociation, and the sulphur is separated as such; the author is engaged in further investigating this subject.

*55. "Monochloridiparaconic Acid and some Condensations." By HENRY C. MYERS, Ph.D.

An attempt to prepare methylparaconic acid by the reduction of the trichloro-acid furnished the dichloro-acid, which on treatment with barium hydroxide suffered condensation, forming the acid $\text{C}_9\text{H}_9\text{ClO}_2$, which has been called monochloridiparaconic acid; its constitution is under investigation. This acid loses its chlorine on treatment with nascent hydrogen, producing a compound having in all probability the formula $\text{C}_9\text{H}_{12}\text{O}_2$, but it is so unstable that its investigation is very difficult. These condensations are being further investigated.

56. "Corydaline." Part V. By JAMES J. DOBBIE, M.A., D.Sc., and FRED MARSDEN, M.Sc., Ph.D.

When corydaline is heated on the water-bath with very dilute nitric acid (about 1 : 20), a difficultly soluble nitrate, $\text{C}_{22}\text{H}_{29}\text{NO}_4 \cdot \text{HNO}_3$, is first formed.

On further heating, the solution becomes dark red in colour, and soon ceases to give any precipitate on testing with ammonia. If at this stage the solution be allowed to cool, groups of bright yellow prismatic crystals separate out on the sides of the vessel. The crystals consist of the nitrate of a base—dehydrocorydaline—differing from corydaline by four atoms of hydrogen (*cf. Trans.*, 1897, lxxii., i., 175). The free base is very soluble in water and alcohol, and is difficult to obtain in crystals. The nitrate, $\text{C}_{22}\text{H}_{25}\text{NO}_4 \cdot \text{HNO}_3$; the chloride, $\text{C}_{22}\text{H}_{25}\text{NO}_4 \cdot \text{HCl}$; the platinumchloride, $(\text{C}_{22}\text{H}_{25}\text{NO}_4 \cdot \text{HCl})_2 \cdot \text{PtCl}_4$; and the chloroform compound, $\text{C}_{22}\text{H}_{25}\text{NO}_4 \cdot \text{CHCl}_3$, are described. The solutions of dehydrocorydaline and its salts have an intense yellow colour, and give a vivid green colour with blue litmus. Reducing agents re-convert dehydrocorydaline into optically inactive corydaline.

If concentration of the acid solution be continued beyond the point at which dehydrocorydaline is formed until platinum chloride no longer gives a precipitate, yellow coloured crystals of an acid melting at 218° separate out on cooling. This acid, to which, for convenience of reference, the name *corydic* is temporarily given, is readily soluble in hot water and in alcohol, but insoluble in ether. Its aqueous solution has an intense yellow colour, and does not give precipitates with any of the metals in aqueous solution.

A silver salt, $\text{C}_{18}\text{H}_{15}\text{NO}_6\text{Ag}_2$, is obtained by precipitating an alcoholic solution of the potassium salt of the acid with an alcoholic solution of silver nitrate. Corydic acid contains two methoxy-groups and is dibasic. Its formula is $\text{C}_{14}\text{H}_9\text{N}(\text{OCH}_3)_2(\text{COOH})_2 \cdot \frac{1}{2}\text{H}_2\text{O}$. When heated with hydrogen iodide it yields, a highly insoluble phenolic acid, $\text{C}_{14}\text{H}_9\text{N}(\text{OH})_2(\text{COOH})_2 \cdot 2\text{H}_2\text{O}$, which separates from a large quantity of hot water in brilliant yellow spangles. The lead salt of this derivative, dried at 130° , has the composition $\text{C}_{16}\text{H}_{11}\text{NO}_6 \cdot \text{Pb}$.

When corydic acid is boiled with a solution of potassium permanganate, it yields a mixture of at least four acids.

(1). An acid, $\text{C}_{12}\text{H}_6\text{N}(\text{OCH}_3)_2(\text{CO}_2\text{H})_3$, which crystallises from hot water in small white acicular crystals melting at 228° . This acid, which is the chief product of the oxidation, contains two methoxy-groups. A silver salt, having the composition $\text{C}_{17}\text{H}_{12}\text{NO}_8\text{Ag}_3$, has been prepared. (2). A hemipinic acid, yielding an ethylimide which melts at 228° , and differs therefore from the hemipinic acid previously described as occurring amongst the

products of oxidation of corydaline with potassium permanganate. It is thus established that corydaline contains two benzene nuclei, and the formation of corydic acid from the alkaloid is easily explained on the assumption that one of the rings is oxidised. (3). A nitrogenous acid melting at 208° which contains no methoxy-groups and gives a faint brownish yellow colour with ferrous sulphate solution, and evolves a strong odour of pyridine when heated with lime. This acid has the formula $C_9H_7NO_6 \cdot H_2O$, and is apparently a methylpyridine tri-carboxylic acid. It is not identical with any of the known acids of this composition. It forms a silver salt, $C_9H_4NO_6Ag_3$, and gives precipitates with lead, barium, and copper. (4). A nitrogenous acid melting at 243° , which gives a yellowish red colour with ferrous sulphate and contains methoxyl.

Oxalic acid and the nitrogenous acid melting at 208° were obtained from the mother-liquors of corydic acid.

Ordinary Meeting, May 6th, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

CERTIFICATES were read for the first time in favour of Messrs. William Ackroyd, 9, Grandsmere Place, Halifax; William Malam Brothers, Beechwood House, Prestwich, near Manchester; William Cranfield, 5, Second Avenue, Halifax; A. F. Bilderbeck Gomez, 24A, Alfred Place West, South Kensington; Frederick Roscoe Grundy, B.Sc., 20, Derby Road, Douglas, Isle of Man; Edward Halliwell, Alexandra Crescent, Dewsbury; Harold Harman, Brewers Sugar Co., Greenock; William Robertson Pollock, Kirkland, Bonhill, Dumbarton.

THE PRESIDENT stated that, in accordance with the instructions of the Council, the two Scrutators, in the presence of the Secretaries, had re-counted the votes recorded for the Presidency at the Anniversary Meeting. The Scrutators' report will be presented to the Council in due course, but he thought he might now venture to say that he learned from the Senior Secretary that the votes accepted by the Scrutators were 166 for the Council's nominee, and 152 against.

The following were duly elected Fellows of the Society:—Messrs. Herbert W. L. Barlow, M.A., M.B.; William Barlow; Alfred Hunter Boylan; James Brierley; Henry Norris Davidge; Louis Charles Deverell; Alexander Duckham; Alaric Vincent C. Fenby, B.Sc.; Charles Henry Field; Thomas Girtin, B.A.; R. Glode Guyer; Harold William Harrie; Sydney Hill; W. J. G. Lasseter, M.A.; Charles Macculloch; Willie Lee Mallinson; George Fowle Merson; Edmund Howd Miller, M.A., Ph.D.; Tom Mitchell; Frederick Filmer de Morgan; Joseph Previte K. Orton, B.A., Ph.D.; Harry E. W. Phillips, B.A.; Robert Howson Pickard, B.Sc.; Thomas Tickle; William Herbert Waite, B.A.; Charles Thomas Foster Watts; John Welsh; Charles Alfred West; Paul Thomas White.

Of the following papers those marked * were read:—

*57. "A Bunsen Burner for Acetylene." By A. E. MUNBY, M.A.

The cheap production of calcium carbide has placed a powerful illuminant within the reach of those who possess no gas supply, but so far little has been heard of the use of acetylene as a heating agent. Our laboratory is, as far as we know, the first to make use of the gas for this purpose. We employ a Bunsen burner of special dimensions, the tube being 5 m.m. in internal diameter. A slightly wider tube may be used, provided the mouth be curved inwards, so that the actual exit does not exceed the diameter mentioned; if larger, the flame tends to strike down. The gas jet is very small, being only capable of delivering about 1 cubic foot of acetylene per hour under 6 inches water pressure, such a rate of con-

sumption giving an ordinary working flame. The air-holes and collar are arranged as in an ordinary Bunsen, the exact size of the former not being of much importance provided they be large enough to admit the air required. The burner is protected with a cap, when not in use, as its efficiency depends upon the jet maintaining its dimensions. A generator capable of giving gas under 7 inches water pressure with the full number of burners in use is required. Under this pressure a large, perfectly blue flame is obtained, which may be turned down to what may be termed a quarter Bunsen flame, equivalent to burning the gas under 3 to 4 inches water pressure. This is the smallest pressure with which the burner will give a non-luminous flame; when turned lower, the zone of partial combustion appears, since the draught is then insufficient.

The heating effect of the flame is of course very great, enabling one to dispense with the blowpipe for some operations, such as small fusions. From a few experiments on heating equal quantities of water under like conditions with coal-gas and acetylene, it would seem that in practice, for equal volumes burnt, the latter has nearly twice the heating power of the former.

The use of the gas should do much to stimulate research in country places and on private estates.

*58. "The Reactions between Lead and the Oxides of Sulphur." By HENRY C. JENKINS and ERNEST A. SMITH. (This paper appeared in our last issue, p. 241).

DISCUSSION.

Professor ROBERTS-AUSTEN said that he was much gratified by the fact that the accuracy of the time-honoured equations given by his distinguished predecessor, Dr. Percy, as representing the metallurgy of lead, had been abundantly justified by work done in the Metallurgical Laboratory of the Royal College of Science. He could testify to the extreme care which Messrs. Jenkins and Smith had given to the work, and the reversible reactions they had discovered were not only very interesting, but of much industrial importance. In justice to Mr. Hannay, it might be conceded that the singular nature of the reactions discovered by the authors of the paper justified Mr. Hannay in questioning the accuracy of the old equations, and it was satisfactory that the difficulty had now been solved.

THE PRESIDENT, Mr. GROVES, and Prof. ARMSTRONG thought the authors' experiments showed that it was unnecessary to suppose that any new compound of lead and sulphur was concerned in the metallurgical process.

*59. "X Ray Photographs of Solid Alloys." By C. T. HEYCOCK, F.R.S., and F. H. NEVILLE.

In a previous communication (*Trans.*, 1889, lv., 666) we discussed the behaviour of gold in sodium and the state of the gold in the solid alloy. We have lately examined alloys of gold and sodium by cutting thin sections from cylinders of the alloy of various concentrations, placing these on a photographic plate, and photographing them by means of the X rays.

On account of the much greater transparency of sodium to these rays as compared with gold, the individual crystals of the alloy are clearly shown. The plates were about 12 m.m. thick, and the alloy had been allowed to cool and solidify very slowly. Pure sodium shows no crystalline structure, but an alloy containing 3 per cent of gold shows a mass of transparent sodium crystals, with dark spaces between the crystals where the gold has concentrated during the process of solidification.

A 10 per cent alloy of gold shows the same phenomena, but the crystals of sodium are narrower, and the dark spaces occupy a larger area.

The appearance of the sodium crystals strongly recalls the fern-like pattern seen when ammonia chloride is crystallised on a microscopic plate, doubtless due to the fact that sodium, like so many other metals, crystallises in the regular system. An eutectic alloy of gold and so-

dium (23·1 per cent gold), shows, as one would expect, scarcely any structure, because the crystals of gold and sodium separate out simultaneously, and are too minute to be detected by such a method.

A solution obtained by saturating sodium with gold at a temperature considerably above the melting point of sodium shows a net-work of black, opaque needles, which are no doubt crystals of gold which have separated and grown to a considerable size as the liquid cooled.

No sodium crystals are here visible, as the groundwork consisted of the eutectic alloy. We have been able to demonstrate the internal structure of a solid alloy, and to show that the process of solidification is strictly comparable to that of a saline solution, the details being perfectly visible to the naked eye.

We have already examined some aluminium alloys by this method, and hope to present a complete account of the work to the Society. The method will probably be applicable in all cases where there is a considerable difference in transparency between the metals of an alloy.

DISCUSSION.

Prof. ROBERTS-AUSTEN said that the use of Röntgen rays for revealing the structure of certain alloys possessed advantages when it was desirable to view the alloys as transparent, as distinguished from the structure shown by a section in a single plane. The structure of alloys as revealed by microphotography had now attained great perfection, notably in the hands of M. Osmond in France, and it was a subject to which Professor Austen had recently given much attention; but the X rays might be very useful in the case of alloys containing one transparent metal. When Röntgen's great discovery was first published, Prof. Austen had examined the relative transparency of certain metals, and endeavoured to detect the difference between hard and soft steel, but the sections of steel employed were too thick, and the experiments were abandoned.

It had been shown by Osmond and by Charpy that eutectic alloys have a pearly structure, and Prof. Austen thought that the gold-sodium eutectic shown on the screen by Mr. Heycock also had the pearly structure. As a good example of the distribution produced by freezing, Prof. Austen stated that if a triple alloy of copper, antimony, and lead was cast as a rod and fractured transversely, a purple ring of the "regulus of Venus" (the copper antimony alloy) surrounded the grey alloyed lead which was driven to the centre, so that there was a grey rod inside a purple tube. He hoped that Messrs. Heycock and Neville, who were so greatly extending our knowledge of alloys, would continue their investigations.

The PRESIDENT remarked that this was an important and interesting communication, as illustrating the service the Röntgen rays might be to the chemist. Soon after the announcement of the original discovery, he made experiments on the transparency of the elements to the new rays, and took a number of photographs. In a verbal communication to the Royal Society, he had announced that opacity of elements to the rays increased in the same series with their atomic weight. He thought the rays might sometimes be of use in settling doubtful cases of atomic weight. For example, if liquid or solid argon were found to be less transparent than oxygen or nitrogen in a similar condition, it might be safely concluded that its atomic weight is higher than that of either of the other constituents of air.

Research Fund.

A meeting of the Research Fund Committee will be held in June. Applications for grants, accompanied by full particulars, should be sent to the Secretaries before June 8th.

List of Fellows.

A new list of Officers and Fellows of the Chemical Society being in course of preparation, it is requested that Fellows will send any alteration of address, without delay, to the Assistant Secretary, Burlington House, London, W.

NOTICES OF BOOKS.

Tea: a Text-book on Tea-planting and Manufacture.

Comprising Chapters on the History and Development of the Industry, the Cultivation of the Tea-plant, the Preparation of the Leaf for the Market, the Botany and Chemistry of Tea, &c.; with some Account of the Laws affecting Labour in Tea-gardens in Assam and elsewhere. By DAVID CROLE (late of the Jokai Tea Company, &c.). Illustrated. London: Crosby Lockwood and Son, Stationers' Hall Court, Ludgate Hill. 1897. 8vo., pp. 242.

WE have here an encyclopedic monograph on tea, almost from every conceivable point of view. The planter, the merchant, the general consumer may all very well be interested in a natural product which is here truthfully pronounced as the great "rival of alcohol" among the British race, and which unfortunately shares its deleterious action on public health. The "tea-dinner" is a powerful agent in sapping our national health and vitality, by the simple process of rendering animal foods of various kinds indigestible, and by the equally unsatisfactory trick of substituting "bread and butter" for more nourishing viands. But our author, whilst fully admitting the perils of tea-drinking in excess, as indulged in by Samuel Johnson, lays down three conditions under which the Indian herb may be consumed not merely with safety, but even with benefit. He tells us not to use water which has been boiling before. "Put cold water in the kettle, and directly it comes to a boil use it for making the tea, and also for previously heating the tea-pot and cups."

"Infuse for about four minutes, and five at the outside, and then drain the liquor into another vessel, so that no tea-leaves remain soaking in the liquor."

"It is best, from every point of view, to use only Indian or Ceylon tea."

These rules substantially agree with the practice of experienced tea-brokers, but unfortunately they disagree with many laundresses and other poor women, who allow a minute dose of tea to simmer on the hob until not only all the tea but all the tannin have been extracted. Mr. Crole refers to the very extensive use of non-narcotic beverages,—tea, maté, coffee, cocoa, &c.,—and suggests that they are in some unknown method utilised in the human economy. To solve this and similar questions would doubtless be a worthier expenditure of brain-power than preparing for examinations.

In Assam the greater part of the tea crop grows in the rich soil of the valleys. A bush to do its best requires about 16 square feet of well-cultivated soil around it. The tea-plant requires a very rich soil, a tropical rainfall such as, e.g., 10 inches daily, and a very hot sun.

The author thinks that not only British colonies, but Russia, are preferring Assam tea to those imported overland from China. Without this increasing demand the tea-trade of India and Ceylon will be swamped by overproduction. The author suggests that it would be a wise policy if the tea-growers would seek to open out new markets. At present they are chiefly following out the suicidal policy of opening up fresh lands. One of the perils of the planter must be sought in the inroads of stray beasts, buffaloes, and — what is much worse — elephants, unless the prepared lands are fenced in with barbed wires.

Among the animal parasites which work havoc in a plantation is the "red spider," not unknown in our conservatories at home.

The mosquito blight is a scourge incorrectly named, but fatally destructive. The only remedy known is to burn down, root and branch, all the bushes affected. Mr. Crole recommends the most jealous inspection, lest the enemy should, like the coffee-bug of Ceylon, suddenly overwhelm all the plantations. The number of possible anti-

dotes, and the season and manner of their application, require careful study. Copper sulphate has been tried for "mosquito blight," but not with sufficient perseverance to admit of a decisive judgment. We hope that some experimentalist will appear more successful than Dr. Koch has proved against the *Rinderpest*.

In "Appendix B" we find a list of blight, microbia, and their supposed remedies and their enemies,—proved or hoped for,—which will prove suggestive reading.

The aspirant after laurels, in this most useful or rather necessary warfare, must bear in mind that what will destroy an animal may be absolutely harmless to a parasitical plant, and *vice versa*.

The operations of withering and drying the tea-leaf have given scope to no little skill, and the results, those especially of the Gibbs machine, leave little if anything to be desired.

Mr. Crole remarks it as an inconsistency that, after the utmost care has been used in every detail of the tea-manufacture, it is stamped into the boxes with the boot-shod feet of the people engaged in the warehouses in London.

Mr. Crole's work must commend itself to the favourable notice of a vast and influential portion of the British public.

Respiratory Proteids, Researches in Biological Chemistry.

By A. B. GRIFFITHS, Ph.D., F.R.S. (Edin.), F.C.S., Lecturer on Chemistry at the Brixton School of Pharmacy, Member of the Chemical Societies of Paris and St. Petersburg, Author of "Physiology of the Invertebrata," "A Manual of Bacteriology," "Researches on Micro-organisms," &c. London: L. Reeve and Co. 1897. Pp. 126.

THE purpose of the present work is to expound recent researches in biological chemistry, giving details of the respiratory proteids in the blood of animals. The author concludes that the blood of the earthworm is chemically comparable to that, *e.g.*, of the dog. The blood of insects (*e.g.*, the house-fly) contains hæmoglobin. The blood of the Lepidoptera is principally green. Lepidoptera are free from respiratory pigments, but have still a respiratory function. According to Regnault, Reiset, and Munk, insects, in proportion to their weight, take up as much oxygen as the highest vertebrates. Poulton has shown that larval blood coagulates much more rapidly than pupal blood.

The blood of three species of arachnids has been analysed, and found in every case to contain copper in the form of hæmocyanin. The blood of most gasteropods and cephalopods also contain hæmocyanin.

The spectra of certain respiratory pigments (*e.g.*, echinochrome, oxychlorocruent, oxyhæmoglobin, hæmoglobin, myohæmatin, alkaline hæmatoporphyrin, and acid hæmatoporphyrin) are shown in a diagram; the serum shows two bands in the green, but turns to a violet-red if allowed to remain in contact with the clot. Chlorocruorin, a green colouring-matter found in the blood of *Saballa*, is a respiratory pigment.

Hæmocyanin, the chief oxygen carrier in the blood of the higher invertebrates, may be conveniently studied in the blood of the lobster, the crab, and the sepia, which contain it to the extent of about 0.30 per cent. Hæmogonin gives a characteristic rose-colour with Millon's reagent. Pinnaglobine contains manganese. The author points out that the respiratory products of animals (Mn=55, Fe=56, Cu=63) do not differ much from each other, and asks if these weights have any important meaning. "What bearing has Prout's hypothesis or Crookes's theory of the genesis of the elements on the subject? Why should the methods present in respiratory proteids have practically the same atomic weight, and be located about a quarter of the distance from the lowest weight (H=1) to the highest weight (U=240)? Has the subject any bearing on the doctrine of evolution, the

theory of natural selection, and the action of the environment?"

These are questions which merit and will doubtless repay a careful study.

The Appendix contains much interesting matter concerning pelagine, pupine, cupine (containing lithium), carminic acid, lepidotic and lepidopterac acids.

To all students of biological chemistry this little book will prove deserving of close study.

The School of Mines, Laramie, Wyoming. Petroleum Series. Bulletin No. 2, January, 1897.

THIS Report comprises an account of the geology of the Popo Agie, Lander, and Shoshons Oil-fields, by W. C. Knight, Professor of Geology; and an analysis of the petroleum of the same districts, by E. E. Slosson, Professor of Chemistry.

Prof. Slosson remarks that the higher the specific gravity of an oil, the higher will be the flashing, burning, boiling, and freezing points, the darker also the colour, and the greater the viscosity. The specific gravities in this report have been taken at 15° C. by the Westphal balance or the flask. Viscosities have been taken by means of Engler's viscosimeter. The Popo Agie oil is 0.90000, being lighter than samples taken in 1897 and 1887.

The flashing-point of the crude oil is 32° C., and the ignition-point 58° C. It is still fluid at -10° C. Its calorific power, as determined by the bomb-calorimeter, is 10,437 calories per grm. = 14,571,000 foot-pounds of energy per lb. of oil. It is not fluorescent, and on distillation light oils and non-condensable gases are given off at 300°—32°, and paraffin appears in the heavier distillates.

In its crude state Popo Agie oil is inferior to the Salt-creek oil, on account of the presence of light oils, tar, and sulphur compounds.

All the products of the distillation of the Popo Agie petroleum can be capable of utilisation. The gases can be used for firing, and the coke, which is very hard and porous, is fit for metallurgical uses. The residues from filtration can be worked up into paraffin or vaseline.

Report of the Senior Analyst of the Department of Agriculture of the Cape of Good Hope, for the Year 1896. Cape Town: W. A. Richards and Sons. 1897.

THE number of articles analytically examined and reported on during this year is lower than in the year immediately preceding, but is nevertheless very satisfactory when compared with the work of former years, being almost double that of the previous highest.

Among the foods analysed under the Adulteration Act, we note that that 21 per cent were found to be adulterated in 1896, as compared with 18 per cent and 30 per cent during 1895 and 1894 respectively. These figures show the great necessity existing for proper control over the quality of foods offered for sale; milk and coffee were the greatest sinners (or, perhaps, sinned against) in this respect.

As usual, a number of samples of water have been submitted for analysis to ascertain their suitability for drinking purposes, but no very definite results are here recorded. "In my last three annual reports," we read, "reference was made to the bad quality of practically all the water obtained from wells at Rondebosch and Claremont. Each year brought its evidence in the direction of pollution, and 1896 has not been exceptional in this respect. Again have samples of well water from Rondebosch been submitted for analysis, and again have these waters been found to be polluted and unfit for use." This points to a serious lack of control; if no steps can be taken in four

years to remedy such a state of things, is it to be wondered at, that the death-rate from typhoid and fevers is so high in South Africa?

A most important matter in a large agricultural country like Cape Colony is the analysis of soils, and the recommendation of the most suitable fertilisers to be added. Eighty-nine of such analyses have been made over a widely extended area.

CORRESPONDENCE.

PERMEABILITY TO X RAYS.

To the Editor of the Chemical News.

SIR,—In an article on the "Permeability of Various Elements to the Röntgen Rays" published in the CHEM. NEWS of December 18th, 1896 (vol. lxxiv., p. 291), I mentioned an experiment with sodium fluoride, chloride, bromide, and iodide, with a view to seeing how the permeability of the halogens varied with atomic weight. Unfortunately the coil used gave such a small spark that the sodium fluoride alone showed any permeability. Since then I have obtained a larger coil and have been able to repeat the experiment. Instead of sodium iodide, however, I took the equivalent quantity of iodine. It was quite evident that fluorine is much more permeable than chlorine, which latter is more permeable than bromine or iodine, these two being nearly alike. Amorphous phosphorus and sulphur tested at the same time were somewhat more permeable than sodium chloride containing the same weight of chlorine, but considerably more permeable than sodium fluoride. On the same plate I had the radiograph of a crystal of beryl, 12 m.m. thick, and of a crystal of zircon of half the thickness. The latter was practically opaque, the former very perceptibly permeable. (Beryllium has a low atomic weight and zirconium a high atomic weight; garnet and zircon are, I think, the only gems containing elements of high atomic weight, and they are the only impermeable ones). I have now tested the impermeability of all the elements whose atomic weight is not above 40, and of a very considerable number of those with high atomic weight.

All the elements with high atomic weight below that of phosphorus are a great deal more permeable than those of atomic weight higher than potassium, those elements with atomic weight between 30 and 40 filling in the transition space somewhat imperfectly.

The atomic weight of boron differs from that of aluminium by just the same amount that fluorine does from chlorine, but the permeabilities do not differ nearly so much in the former case as in the latter. There is always a great difference between the permeability of an element with atomic weight below 30 and a similar element whose atomic weight is above 30.—I am, &c.,

JOHN WADDELL.

Royal Military College,
Kingston, Canada.

ESTIMATION OF CARBON IN FERRO-CHROME.

To the Editor of the Chemical News.

SIR,—In your last issue appears the record of a very valuable series of experiments carried out by Messrs. Brearley and Leffler on the "Estimation of Carbon in Ferro-chrome." The authors, however, assert that my method is only capable of yielding about $4\frac{1}{2}$ of the 9 per cent of carbon present in such alloys. Such a statement should only have been made on the surest grounds. As a matter of fact the method when properly carried out yields on an average 8 to 9 per cent of carbon as the contents of rich ferro-chromes, and that without the violent reactions described by the authors.

They also say "no naked tube obtainable would stand the ordinary furnace heat." It is true that such tubes are not easy to obtain, but they are nevertheless obtainable.

It would be interesting to know the type of furnace used by the authors, also the diameter of the main gas-pipe. With a 2 inch gas main, a Hoffman furnace in which the openings of the clay burners have been enlarged, and with the gas at night pressure, a blowpipe is not necessary for the combustion of 0.5 grm. of ferro-chrome flour with 10 grms. of chromate of lead. With a poor furnace, or a limited gas supply, the results are as described by Messrs. Brearley and Leffler. With reference to the authors' statements about moist and dry oxygen, it is evident that they do not quite grasp the meaning of these terms. It is also clear from their remarks that they have never used dry oxygen in which bright steel drillings are unoxidised at a full red heat. My instructions had reference to almost chemically dry oxygen issuing from a tube closely packed with minute granules of potash-pumice, previously well dried. To restore the necessary humidity to the gas it was passed over "some loose fragments of slightly moistened asbestos."

It is obviously impossible that gas taking up moisture from shreds of damp asbestos placed at the cool inlet end of the tube could deposit water at the hotter exit end. Moreover, I never obtain a deposit of "oxide of lead" in the position just named, for the simple reason that between the ferro-chrome and the exit there are three plugs of asbestos and a 6-inch column of closely packed small fragments of copper oxide.—I am, &c.,

J. O. ARNOLD.

The Technical School, Sheffield,
May 24, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société Chimique de Paris.

Series 3, vols. xvii. and xviii., No. 8.

Study of the Hydration of Meta-phosphoric Acid.

—MM. Berthelot and André.—The authors show that sodic meta-phosphates prepared—(1) By heating monosodic phosphate to 280°; (2) by melting this salt at a red heat—behave very differently in dilute solutions. The acid titration of the first salt, done immediately after its solution, and again after the lapse of a variable number of days, indicates the presence of a considerable quantity of a mixture of ortho- and pyro-phosphoric acids, while an acetic magnesia mixture produces, when warmed, an abundant precipitate of ammonia-magnesian pyrophosphate, representing 80 per cent of the total phosphorus present. The acid titration of the second salt, on the contrary, indicates that, under the same conditions, the formation of ortho- and pyrophosphoric acids is much slower; the precipitate only representing 50 per cent of the total phosphorus. The free acids prepared by means of these salts confirm these results.

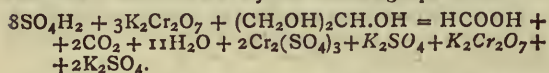
Remarks on the subject of Colour Reactions of Quinine.—E. Blaise.—The Vogel reaction is independent of ferrocyanide of potassium; it suffices to treat the quinine salt with bromine-water and dilute ammonia to obtain the red colour; this becomes green if strong ammonia be used.

Law of Thermic Constants.—D. Tommasi.—A large number of measurements of the heat of combination have been made since 1882 confirming the views then put forth by M. Tommasi. Many objections were at first raised, but these have been finally swept on one side and satisfied.

Cause of Oxidation of Imitation Gold Foil.—Leo Vignon.—Not suitable for abstraction.

Research on the Coal-tar Colours in White Wines, and the Difference between these Colouring Materials and those of Caramel.—A. D'Aguiar and W. da Silva.—It has been suggested that the methods used to detect coal-tar colours in matured wines is at fault, and causes confusion of caramel with aniline, yellow and orange. The authors give the results of three series of experiments made by them with eleven aniline colours, one caramel, and one natural wine. They conclude that caramel, treated in the ordinary manner by amyl alcohol made alkaline with ammonia, gives results always very doubtful and sometimes negative. The yellow aniline colours, on the contrary, give very clear results. Experiments with amyl alcohol in barytic solution, and acetic ether in barytic and ammoniacal solution, gave no better results with regard to caramel. Further experiments were made with subacetate of lead, acetate of mercury and potash, and acetate of mercury and magnesia, and the authors finally conclude that the methods commonly in use may be in every way relied on.

Estimation of Small Quantities of Glycerin.—M. Nicloux.—The estimation of glycerin, based on the reaction of bichromate of potash and sulphuric acid, has been studied by many chemists. The author traverses the statement of MM. Bordas and Raczkowski that the reaction can be formulated by the following equation:—



The mistake, he says, cannot be accounted for by a printer's error, as on one side of the equation we have 3 molecules of bichromate, and on the other only 1 molecule of bichromate and sulphate of potassium.

Notice to Authors.—The Secretary begs that the authors of any papers (French or foreign) sent in to the Society will also send an abstract. This is rendered necessary by the large number of communications received, and the fact that the salient points may be unintentionally overlooked by reason of the abstractor not being so well versed in the particular subject as the author must be. It has been decided that such abstracts, when signed by the author, will be printed at the earliest opportunity, if not more than one page in length, and if sent in not later than a fortnight after the publication of the original.

MISCELLANEOUS.

Atomic Models (Patent No. 1999, 1897).—A patent has recently been granted to Mr. Frederick George Edwards, of London, by which the Government afford protection to his idea that all atoms can be represented by varying numbers of tetrahedrons. The germ of the idea appears to be that as there about seventy elements known to chemists, and that tetrahedrons can be grouped together in as many as seventy ways, the latter can illustrate the former. This is the idea; the practice, the inventor shows, is not so simple. For instance, he says:—"Regular tetrahedrons do not fit exactly, but each tetrahedron is so nearly regular that it may be supposed each of the elements were (*sic*) created from regular tetrahedrons in a plastic condition." This strikes us as a beautiful example of inventing facts to fit a theory. We are glad to find that Mr. Edwards has not patented atoms *per se*, but merely the form he thinks they take, together with a few names of elements, hitherto undiscovered, but predicted by him. These are:—Icosagon, atomic weight 10; x-odine, atomic weight 215; and zadmium, atomic weight 245. It will be interesting to watch the action

for infringement of patent which will result if any chemist engaged in research should have the temerity, or the misfortune to discover either of these predicted elements. A lithographed diagram at the end gives the *shape* of thirty-two elements, with atomic weights made to fit; we are sorry to have to record the fact that many of these atomic weights are wrong, but then so probably are the shapes.

NOTES AND QUERIES.

* * Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Pile Oil.—Can any correspondent inform us who are the manufacturers or proprietors of a drug or preparation known as "Pile Oil."—A. O.

MEETINGS FOR THE WEEK.

TUESDAY, June 1st.—Royal Institution, 3. "The Heart and its Work," by Dr. Ernest H. Starling.

THURSDAY, 2nd.—Chemical, 8. "Thermo-Chemistry of Carbohydrate Hydrolysis," "Thermal Phenomena attending the Change in Rotatory Power of Freshly Prepared Solutions of certain Carbohydrates, with some Remarks on the Cause of Multirotation," by Horace T. Brown, F.R.S., and Spencer Pickering, F.R.S. "Optical Inversion of Camphor," "Derivatives of Camphoric Acid—Part II. Optically Inactive Derivatives," "Racemism and Pseudoracemism," by F. S. Kipping, Ph.D., D.Sc., and W. T. Pope. "On Some New Gold Salts of the Solanaceous Alkaloids," by H. A. D. Jowett, D.Sc.

Royal Institution, 3. "William Godwin and Mary Wollstonecraft," by Churton Collins, M.A.

FRIDAY, 4th.—Royal Institution, 9. "Signalling through Space Without Wires," by W. H. Preece, F.R.S.

SATURDAY, 5th.—Royal Institution, 3. "Music in England during the Reign of Queen Victoria," by J. A. Fuller Maitland, M.A.

IMPORTANT NOTICE.

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1958.

ON THE INTERVENTION OF MANGANESE IN THE OXIDATIONS INDUCED BY LACCASE.

By GABRIEL BERTRAND.

As I have already had occasion to remark, the soluble oxidising ferment of the lac-tree laccase yields, on incineration, an ash relatively rich in manganese oxide.

On combining the use of the colorimeter with Hoppe-Seyler's reaction, which consists in converting the manganese into permanganic acid by lead binocide and nitric acid, I have found that 1 grm. of laccase extracted from Armamase lac contains—

Water of hydration	0.072
Ash	0.046
Manganese	0.0017

that is to say, a proportion of manganese close upon 2.5 per cent of the weight of the ash.

I have since submitted an aqueous solution of this laccase to a fractionated precipitation by alcohol, and I have thus obtained two novel ferments, one of which is more active and the other less active than the original laccase. But on comparing these samples with each other, I observed that their oxidising power varied in the same direction as their proportion of manganese.

Thus the volume of oxygen fixed in ninety minutes by 50 c.c. of solution of hydroquinone at 2 per cent, in presence of 0.260 grm. of the product supposed to be dry, has been :—

Sample No. 1	19.1 c.c.
„ No. 2	15.5 „
„ No. 3	10.6 „

whilst the determinations of manganese gave respectively—

No. 1	0.159 per cent
No. 2	0.126 „
No. 3	0.098 „

Is this a simple coincidence, or is the activity of the soluble ferment due to the presence of the manganese? It seemed important to establish this point.

To this end I first sought to eliminate all the manganese of the laccase at my disposal. But the problem being doubtless too delicate, I have not yet been able to solve it in a satisfactory manner. In some cases the elimination of the metal was too incomplete, and in others the reagent modified at the same time the organic matter.

Fortunately I had another method. It is known that laccase, or at least substances closely allied to it, are found in the generality of green plants. I have therefore extracted laccase from a series of different species, varying a little the method of operation, and probably by reason of a special composition of the cellular juice, I succeeded in obtaining from lucerne a product very poor in manganese, and having little activity under these conditions, but which resumes its activity on the addition of a minimum quantity of a salt of manganese. The product is prepared as follows:—Several kilos. of ordinary lucerne (*Medicago sativa*), gathered at the beginning of the flowering season, were at first bruised and submitted to the press. The juice, saturated with chloroform, was left to coagulate in the dark. After twenty-four hours the juice was filtered,

2½ vols. of alcohol were added to the precipitate, drained, and taken up in a little water. It gave on filtration a limpid liquid of a pale yellow, from which an excess of alcohol (about 5 vols.) separated flocks nearly white, easy to collect, and which rapidly dried *in vacuo*.

This specimen of laccase, extracted from lucerne, contained then—

Water of hydration (determined at 110°)	12.4 %
Organic matters	42.4
Ash	45.2
And a very small proportion of manganese, less than 1-50,000th.	

On dissolving it, in the proportion of 0.100 grm. in 50 c.c. of solution of hydroquinone, we observe, even after twenty-four hours of continuous agitation in contact with air, merely a red colour. If, on the contrary, we add to the same solution 1 m.grm. of manganese (*e.g.*, in the state of sulphate), in about two hours there appear in the first crystals of quinhidrone evident signs of oxidation.

The experiment may also be conducted quantitatively. We then operate according to the method already described, and measure the oxygen absorbed. We thus find for the proportions above given, and a uniform agitation of six hours, at the ordinary temperature (about 15°):—

1. With manganese alone (check experiment) 0.3 c.c.
2. With laccase alone (from lucerne), 0.2 c.c.
3. For laccase with manganese, 6.3 c.c.

Manganese cannot be replaced in a useful manner by any other metal, not even iron. I have tried with the proportion of 1 m.grm. of metal, taken as sulphites:—Iron, aluminium, cerium, zinc, copper, calcium, magnesium, and potassium. In no case did the volume of oxygen absorbed exceed a few tenths of a c.c.

These facts show the physiological importance of manganese, and define its rôle in vegetables.—*Comptes Rendus*, cxxiv., p. 1032.

A TROPICAL FOOD.

By JOHN B. COPPOCK, F.C.S., Harris Institute, Preston.

In many parts of the island of Cuba the fruit of the banana tree or its allies is used as the "staff of life" by the natives, and upon it their strength is preserved unimpaired. A sample of a flour made from *Musa paradisiaca*, one of the banana tribe, yielded upon analysis—

Water	10.62
Albumenoids	3.55
Fat	1.15
Carbohydrates	81.67
Fibre	1.15
Phosphoric acid	0.26
Salts other than phosphates	1.60

100.00

We may therefore conclude it to be a starchy food. The nutritive value from a nitrogen point of view is small, but now it is beginning to be recognised that the nitrogenous tissues of the body have less mobility than hitherto assumed, we may consider whether nitrogen ought to occupy the pre-eminent position to which it gets assigned, but rather that less value should be attached to it and more to the carbohydrates, which are great sources of energy.

Now one of the most striking features of this natural product is the solubility of the carbohydrate portion; with only warm water the whole of it forms quickly a thin mucilage which is apparently very digestible.

The extreme solubility of this flour is further emphasized by the fact that it has long been used in the island of Cuba

as a food or gruel for infants just leaving off breast feeding, despite its being essentially a starchy substance.

The phosphoric acid in it is fairly large; hence its value is enhanced when we consider the important part played by phosphorus in the economy.

A microscopic investigation of the structure of the starch grains led to these conclusions. The granules are elongated, fairly elliptical in shape; the layers of the grain are concentric, but only faintly visible; the granules vary much in size, but the large ones show a well-defined hilum.

The flour has the appearance of finely ground oatmeal, but possesses a distinct odour of an agreeable nature.

A few words on the botanical characters. The banana and the tree giving this product belong to the natural order *Musaceæ*. This order is very prolific in the production of fruit; it produces 45 times more fruit per acre than the productive potato and 131 times more than wheat. Of course this action makes it very exhausting to the soil: the soil does not seem to be replenished by artificial manures, but the tree is removed to fresh soil every few years.

M. sapientum is the ordinary banana, the variety giving this flour being *M. paradisiaca*. They are closely related, the two being distinguished by the sweetness, larger size, and succulent nature of the banana.

The young shoots of some species are also eaten after being boiled for food.

THE DISSEMINATION OF MICRO-ORGANISMS, AND THE BEST METHODS OF DESTROYING GERM EMANATIONS FROM SEWER GAS.*

By CHAS. R. C. TICHBORNE, F.C.S., F.I.C., Dep. Pub. Health,
R.C.S.I., &c.

THERE are two characters of germ contagion, which may perhaps be best illustrated in the diseases of scarlatina and enteric fever.

In the scarlatina we know that the disease is largely conveyed by the desquamation.

In enteric fever the germs which are carried by sewer gas are supposed, with considerable force of evidence, to be a fertile source of spreading the disease.

In scarlatina we assume that the "Raft Theory," as Tyndall called it, plays an important part, whilst in the latter we must assume that the contagion is often carried with the vapours which emanate from the drains.

In my present communication I do not propose to deal at any length with the subject of the transmission of germs by the raft theory. It is fairly understood by most physicists and bacteriologists—but I may as well concisely describe it as most scientists understand it. We assume that the ordinary atmosphere, let us say at the sea-level, is largely contaminated with a visible and ponderable matter, which we term atmospheric dust. This atmospheric dust is found largely present in our homes, and also in the streets of populous towns. It consists of ponderable atoms, which, at an altitude of a few feet, almost entirely consists of organic matter. This fact was conclusively demonstrated by Tyndall's beautiful experiments, in producing what he called "optical vacuums," by the combustion of the organic matter. The coarser particles of this atmospheric dust act as kinds of floating rafts, and carry on their surface the finer structures of life. In the case of such a disease as scarlatina, we can easily see how this raft theory plays an important part in the dissemination of the desquamation.

Even the smallest germs that our microscopes have yet revealed have a certain weight. This fact is shown by the absence of germs at high altitudes, such as Mont

Blanc, or in the very still atmospheres, such as is found in the vaults of St. Michau's church. I demonstrated this as far back as 1870, in an "afternoon lecture," delivered at the Royal Dublin Society. I showed that flasks placed all night in the vaults of St. Michau's church were, when sealed next morning, optically empty, or free from atmospheric dust, and were therefore free from germs. We get in these vaults exactly the same result that we should find on the top of Mont Blanc.

I believe this sterility of the vaults of St. Michau's church has been the subject of one if not more papers read before this Academy. I am sure it had escaped the authors' observation that the ground had been already "prospected."

It is worthy of note that the greater part of practical bacteriology is now worked out by an observation, which I think originated with Tyndall,—that cotton-wool is a perfect filter, as regards this atmospheric dust, and that it not only acted upon the coarser particles, but separated the finer micro-organisms.

I may be excused for still further referring to my own researches in this direction, mention of which will be found in the later editions of Parkes' "Hygiene," by De Chaumont. I was able to demonstrate that even the dust at the top of Nelson's pillar contained over 29 per cent of organic matter, and that it was capable of setting up lactic fermentation in a neutral solution of sugar of milk. (Here is a solution of sugar of milk, which, although sterilised, when left in an ordinary room containing atmospheric dust has fermented, and become solid from the formation of calcium lactate).

The composition at every yard that we rise from the ground becomes freer from the ponderous earthy constituents, but richer in germs. Of course we at last come to an altitude where even the micro-organism becomes scarce, and to such regions as the high altitudes of Switzerland, where the germ is unknown. Even on the Mer de Glace the germs may be said to be absent.

So much for the raft theory, which will account for the dissemination of any germs, provided they have arrived at the dry condition or can be attached to a dry particle. What can be more easy to conceive than the spreading of scarlatina by a process such as this? It is almost self-evident.

But there is another mode by which preventable disease is disseminated, which has never to my mind presented such a lucid explanation as regards its propagation. I refer to the theory which supposes that a certain germ—let us say like that accompanying enteric fever, cholera, or such disease, or the poison of yellow fever—are capable of acting as a poison when evolved in sewer gas.

We have an organism which, when carried in water or brought mechanically to a receptive surface, is capable of producing disease, and yet we also find that it is capable of rising in such a vapour as sewer gas. I use the term "vapour," not in the vulgar sense, which assumes a non-permanent gas capable of condensation, but in the sense that means anything flying or escaping off. In this sense atmospheric dust is volatile.

Two special diseases are supposed to arise from the air of sewers or faecal emanations, typhoid fever and diarrhoea; yet if these diseases are caused by bacilli, how are they volatilised, for, however minute, these bacilli are still organic structures? Although a germ is so small that our finest balance will not weigh it, and, although it may only be 0.005 of a m.m. in length, we might as reasonably conceive that the coarser microscopic life—such as Rotatoria or Entomostraca, which we can almost see with our eye—would be volatilised as a vapour, as to conceive that pathogenic microbes would be so transmitted.

Prof. Frankland has shown that liquid sewage matter is not likely to be scattered into the air, except by gas generated in it. He experimented with lithia, a chemical substance, not volatile, but which could be easily detected, and might be said to represent micro-organisms. He

* A Paper read before the State Medicine Section of the Joint Medical Association.

found that no ordinary agitation of the sewer water would produce indications of lithia in the air, but that directly gas began to be generated in the sewage by decomposition the bursting of the gas-bubbles carried lithia into the air (*Proc. Roy. Soc.*, 1877).

Now I think here is the clue to the dissemination of microbes, but not exactly in the direction which he indicated. Such a condition of sewage, *i.e.*, in active fermentation, is hardly conceivable in a general sense in the better constructed drains found connected with houses of a good class,—the houses, in fact, where typhoid seems to luxuriate. We must suppose, however, in a system of town sewage, certain spots in the mains where, from the Frankland cause, the microbes are scattered into the air during fermentation,—or, let us say, violent concussions breaking the sewage into spray,—and then comes the question, How are they disseminated through the whole area of the air space of the system of drains? I can easily conceive that they are there carried by a condensed vapour exactly represented by ordinary dew. At certain hours of the night, just as we see the rising vapour settling as dew in a valley, I believe that the temperature of the water-laden vapour of the sewer is lowered by being met with the layer of cold night air through the open traps, which determine a dew-point in the sewers themselves. I find from actual experiment that the temperature of the sewer water as it flows from the large sewers, or the temperature of the gas in the mains, is generally 2 or 3 degrees above the temperature of the night air. The gas mains bear a somewhat similar position underground to the sewers themselves, and give a very good idea of this variation of temperature. A variation of as much as 10° to 15° may be observed.

Even a sudden rise in the barometer will just determine the deposition of the liquid portions of the gas in the mains, and in the same manner will also determine, even more energetically, a dew line in the sewers. Each particle of dew becomes a raft which will carry microbes upon its surface, perhaps for miles, as long as this "dew" condition lasts, and, as the sun's warmth dissipates the morning dew, the water raft disappears, leaving the microbes suspended in mid air; or, suppose the sewer dew is carried into a warm shaft connected with a dwelling-house, is not the assumption apparent that again we have the water-rafts converted into permanent gas, whilst the now dry germs float about seeking whom they may devour?

I will just conclude by a few remarks upon sewage disinfection from this point of view.

The disinfectants employed in sewage purification can hardly be viewed as actual germicides. The modes in which they are necessarily used create such an amount of dilution that they can only be viewed as retarders of the development of microbes. It is no doubt chiefly from this reason that they are not more extensively used in sewage purification. The return in results, as regards the prevention of diseases, is not commensurate with the great expense of oxidisers such as permanganates and hypochlorites. They are not in favour, because without they are used in overwhelming quantities they are worse than useless. They destroy myriads of microbes, but they allow myriads to escape, and to these they only seem to add fuel to the fire. It is true that at the pumping stations in London manganate of soda is used, or was used; but in that case it is merely employed as a deodoriser at the end of the process, and whilst the supernatant fluid is poured into the river.

From the reason of cheapness, crude carbolic acid (which may be considered to owe its virtue to phenol, cresylic acid, and a little naphthalene) is extensively used. Although not a very decided germicide, phenol still holds an important place as one of the most valuable retarders of germ development. Naphthalene is still more powerful, and may be looked upon as a germicide proper; although very cheap, it has one objection, namely, its insolubility. I have here a fluid which I have used with some success for years, in controlling or instantly stopping

germ development, for which there are many occasions where the use of mercuric chloride is inadmissible. It consists of—

Crystallised phenol	1 part,
Camphor	3 parts,
Naphthalene	$\frac{1}{2}$ part,
Coloured with rosaniline carbolate.	

It will be observed that, though these are all solids, they form a liquid on rubbing together. One drop of this liquid will instantly arrest any tube of microbe culture in gelatin at any point, and may be used with advantage to place on record comparative experiments with microbe cultures.

In such experiments I have found it advantageous to use a little stiffer nutrient gelatin than that given in Crookshank. I increase the formula there given from 100 grms. of gelatin to 120 grms. (*vide* "Manual of Bacteriology," 3rd edit., p. 83).

Now it is a similar preparation to the above which I should propose for sewage purification, with one modification which I consider invaluable. Crude carbolic acid (phenol) is comparatively cheap, and naphthalene may be viewed as a waste product in the process of coal-tar distillation. For the camphor I would substitute terebene, which may be looked upon almost as a liquid camphor. Where the sewers of a large city are being provided for, and where money is a question of importance, the light oils of tar may be substituted.

Now I will describe the scientific theory by which I believe this can be made a trap for typhoid or germs of a like nature in sewer gas.

The principle involved consists in adding some liquid body which shall bring the specific gravity of the antiseptic below that of the sewer matter. When such a body is used the antiseptic forms at once a fine pellicle on the surface of the sewage, and all fluids that are volatilised or mechanically eliminated by the escape of gas must pass through the germicide layer.

If carbolic acid or the crude phenol products are used by themselves, we find in practice that they immediately sink to the bottom of the flowing sewage which passes along over the top in a continuous stream of untouched pollution. The crude product obtained from the distillation of coal-tar is specially suited to this purpose. When coal-tar is distilled, in the first instance, it is divided into two divisions; one is called "light coal-tar oil," the other "heavy coal-tar oil." The first are all the products which come over as long as they will float on water, and they are specially rich in the benzene, naphthalene, and terebene series, all of which are powerful germicides. By substituting these oils for terebene, we get an antiseptic fluid which immediately spreads on the surface of the sewage, locking in the deleterious vapours, and at the same time passing downwards the heavier antiseptics, such as the phenol, by the simple act of solution.

This can be illustrated by the following simple experiment:—If we pass, by a pipette, a layer of carbolic acid into a shallow dish of water, and after standing some time draw off some of the supernatant water, we shall find, on testing it with a little bromine water, that it contains no carbolic acid. If in a second experiment we use such a mixture as I have specified, which has a gravity of 0.850 to 0.950, we shall find, on introducing it into the water with the pipette, that it immediately rises to the surface, and if we at once remove some of the water from the interior, it gives, on testing with bromine water, a copious precipitate, showing that the carbolic acid has permeated at once through the water, which represents the sewage. We should find, on further examination, that the powerful antiseptic naphthalene had been carried with it.

I have endeavoured, in the above experiments, to show why, in many cases, the use of carbolic acid has been a practical failure as a sewage purifier, and to indicate that, as regards dealing with such contagions as are diffused through sewer gas, a principle should be adopted in the

use of antiseptics. This principle has not, so far as I know, been openly enunciated, namely, that we must disinfect from the surface of the flowing sewage, and not from the bottom.

CONTRIBUTION TO THE STUDY OF HYPONITROUS ACID.*

By A. HAUZSCH and A. L. KAUFMANN.

(Concluded from p. 256).

Conductivity of Hyponitrous Acid.

PERFECTLY pure hyponitrite of silver is added in excess to a $1/32$ normal solution of hydrochloric acid at 0° . The $1/32$ normal solution of hyponitrous acid thus obtained is filtered at 0° , and this filtrate is used for the determination of the conductivity.

I. Molecular amount in 64 litres. Temperature 0° .

$\mu^1 = 2.98$	$\mu^5 = 3.65$	$\mu^9 = 4.14$
$\mu^2 = 3.06$	$\mu^6 = 3.78$	$\mu^{10} = 4.24$
$\mu^3 = 3.40$	$\mu^7 = 3.87$	$\mu^{11} = 4.29$
$\mu^4 = 3.46$	$\mu^8 = 3.92$	$\mu^{12} = 4.32$

II. Solution prepared exactly the same as No. I., but kept at 0° for fifteen hours.

$\mu^1 = 1.96$	$\mu^5 = 2.07$	$\mu^9 = 2.18$
$\mu^2 = 1.98$	$\mu^6 = 2.09$	$\mu^{10} = 2.19$
$\mu^3 = 2.00$	$\mu^7 = 2.11$	$\mu^{11} = 2.22$
$\mu^4 = 2.02$	$\mu^8 = 2.14$	$\mu^{12} = 2.25$

The result obtained was similar to that of the first experiment. The initial value was smaller, but it gradually increased.

III. The solution used for the first experiment was examined again after standing for fifteen hours at 0° .

$\mu^1 = 21.65$	$\mu^5 = 23.56$
$\mu^2 = 21.92$	$\mu^6 = 23.68$
$\mu^3 = 21.94$	$\mu^7 = 22.80$
$\mu^4 = 21.83$	$\mu^8 = 23.68$

These experiments show that the conductivity of hyponitrous acid increases with the duration of the current. Under the action of an alternating current, hyponitrous acid ought to give rise to products which would be better conductors than hyponitrous acid itself. As a matter of fact solutions thus treated give, with sulphuric acid, iodide of potassium, and starch, an intense blue colour. Thus we see that the decomposition previously described is effected by the action of an alternate current at 0° .

Experiment III. shows clearly that nitrous acid once formed accelerates the decomposition of hyponitrous acid in a similar manner. As probable equivalent of the conductivity of hyponitrous acid, we must take into account what was observed in the first place. Its value should therefore be, assuming v to be equal to 64, $\mu = \infty 3$.

The determination of the conductivity of very weak solutions, and the calculation of the constants of dissociation, have naturally been impossible to effect with a body which is such a bad conductor and so unstable as hyponitrous acid. But contrary to the general opinion, hyponitrous acid is a very weak acid. It is, in fact, very similar in that respect to carbonic acid, whose conductivity is, according to Knox, $K = 0.0000008$.

Conductivity of Hyponitrite of Soda.

Having proved by previous experiments that the acid hyponitrite of soda, NaHH_2O_2 , was a very unstable compound, we endeavoured to determine the conductivity of the neutral salt. This latter salt could not be prepared

by the direct action of hyponitrite of silver in excess on a solution of chloride of sodium, on account of these bodies acting very slowly and incompletely on one another. We obtained it by mixing equal quantities of hyponitrous acid and caustic soda. The hyponitrous acid was prepared in the ordinary way, by the action of hyponitrite of silver on a normal solution of hydrochloric acid; the caustic soda was prepared according to Hautzsch and Gerilowski's method. The two solutions, mixed at 0° , were diluted so as to contain a $1/32$ normal quantity of hyponitrite of soda.

I. $v = 64$.

$\mu_1 = 111.66$	$\mu_4 = 109.56$	$\mu_6 = 109.26$
$\mu_2 = 110.76$	$\mu_5 = 110.26$	$\mu_7 = 109.16$
$\mu_3 = 109.56$		

II. $v = 126$.

$\mu_1 = 139.06$	$\mu_3 = 135.4$	$\mu_5 = 134.12$
$\mu_2 = 137.06$	$\mu_4 = 134.42$	$\mu_6 = 132.92$

III. $v = 256$.

At first $\mu = 148.04$, after 13 minutes $\mu = 141.14$, after 15 hours $\mu = 126.34$, after 7 minutes $\mu = 143.04$, and after 16 minutes $\mu = 139.64$.

The three series of experiments show that the values of the molecular conductivity diminish from one experiment to another. This can be accounted for in two different ways. We can imagine that the fixation of the carbonic acid in the air, by the strongly alkaline hyponitrite of silver, would have the effect of decreasing the conductivity of the latter. The facts observed in the determination of the conducting power of carbonate of soda show that this supposition is probable. But we may also suppose that, under the influence of the electric current, hyponitrite of soda, strongly hydrolysed, gives rise to products of decomposition of very bad conductivity.

In spite of these difficulties it is easy to see that the conductivity diminishes considerably on dilution. The most probable value is that first observed. With $v = 64$, and $t = 0^\circ$, $\mu = \infty 112$. In any case, however, this high number shows a noticeable splitting up of the disodic salt, easily shown by phenolphthalein.

Comparison of Nitramide with Hyponitrous Acid.

The chemical properties being known, the comparison has been completed by the determination of the molecular weight and the conducting capacity of the nitramide.

The determination of the molecular weight of the nitramide gave the same value as that we found for hyponitrous acid.

Molecular weight: found, 61; theory $\text{H}_2\text{N}_2\text{O}_2$, 62.

Conductivity of Nitramide.

I. $v = 32$; temperature $= 0^\circ$.

Five series of experiments were made, each comprising four determinations. The following figures show the average of each series:—

1. $\mu = 1.97$.
2. $\mu = 1.95$.
3. $\mu = 1.93$.
4. $\mu = 1.93$.
5. $\mu = 1.96$.

II. $v = 64$; temperature $= 0^\circ$.

Means of two series of experiments:—

1. $\mu = 1.69$.
2. $\mu = 1.69$.

These results show that, contrary to the opinion expressed by Thiele (*Zeitsch. f. Physik.*, xvii., p. 185), nitramide is a very weak acid, the same as hyponitrous acid.

The low conductivity makes the determination of the constant of dissociation impossible. But at any rate the values for nitramide ($\mu = \infty 2$) are lower than in the case

* *Moniteur Scientifique*, vol. xi., p. 336, May, 1897.

of hyponitrous acid ($\mu = \infty$ 3). The acidity of the former is therefore slightly more pronounced than that of the latter; but unlike hyponitrous acid, the conductivity of nitramide is not modified under the influence of an alternate current. Nitramide in the free state is thus considerably more stable than hyponitrous acid.

The interchangeability of these two substances, one into the other, by inter-molecular transposition, appeared to be very probable, in view of their isomerism, and the analogy of their proportions which exists; but the numerous experiments undertaken with a view to cause this transformation have up to now given only negative results.

SOME PRESENT POSSIBILITIES IN THE ANALYSIS OF IRON AND STEEL.*

By C. B. DUDLEY.

(Continued from p. 258).

MUCH might be said in regard to the colour test for determining carbon in steel. It is difficult to over-estimate the value and importance of this method, especially in the daily operation of steel works, and there seems little doubt but that if proper precautions are employed, the method in skilful hands will give results that are fairly reliable to within three- or four-hundredths of a per cent. It would hardly be possible in this paper to discuss all the precautions which are deemed essential by those best informed. A chemist of wide experience with the method has enumerated twenty-four points that must be observed, if reliable results are to be expected. Let it suffice for us to say that even approximate accuracy cannot be expected.

1. If the steel whose carbon is to be determined and the standard steel do not have their carbon in the same condition. For example, if the standard steel has been annealed, and the sample to be tested has been tempered, the results will be worthless.

2. If the attempt is made to determine the carbon in any steel by using a standard widely different from it, in carbon content. Using a 0.20 per cent carbon standard, with a steel containing 0.50 or 0.60 per cent, is apt to lead to very fallacious results.

The best results seem to be obtained by having the carbon in all steels both standards and tests in the condition given by annealing, by having a number of standards which differ little from each other in carbon content, and by not attempting to use the method on steels containing very little or very large amounts of carbon. It may not be amiss to add here that the practice so prevalent in many of the steel works, of using this method for all carbon determinations, including those where contracts are involved, is reprehensible, and should be discontinued. The chemist at the works does the best he can with the method he is using, and the amount of work required of him, as well as the facilities furnished, do not admit of the use of a better method. On the other hand, when a dispute arises, and it is ultimately shown that the works are in error, the chemist is blamed and analytical chemistry brought into disrepute, not because either is really at fault, but because more is expected of the colour test method than it is really able to give. To the steel makers we say, "Do not expect your chemists to render you the bricks of good chemical analyses, without you give him the requisite straw of time and appliances to do good chemical work."

Few of the constituents of iron and steel have more important influences on their valuable qualities than phosphorus, and upon few has more chemical work been done.

The present condition of the methods for determining this constituent seems fairly satisfactory, provided we are willing to take time enough to do the work. In confirmation of this statement, the work (*Proc. Am. Soc. Civil Eng.*, xxi., 59) done by the Sub-committee on Methods of the International Committee on Standards for the Analysis of Iron and Steel may be cited. This sub-committee consisted of five members, each of whom analysed five samples of steel, and each used his own method, without any attempt at consultation or agreement with each other before the work was done. The methods employed may be briefly indicated as follows, those interested being referred to the Report of the Committee published as per the reference given for the details. Mr. Blair used what is known as the acetate method. Mr. Shimer used the molybdate magnesia method. Your speaker used a combination of the acetate and molybdate magnesia methods. Dr. Drown used a combination of certain features of the modern rapid methods with the molybdate magnesia method. And Mr. Barba on one sample used the acetate method as described by Blair, and on the other four samples employed certain features of the molybdate method to separate the phosphorus from the iron, and then used the reductor to get the amount of phosphorus, instead of weighing as magnesium pyrophosphate. It will be evident to any one carefully reading the Report referred to that the methods employed differed widely in principle, in strength of solution, and in manipulation, and yet these methods gave the following percentages of phosphorus in the five samples.

	1.	2.	3.	4.	5.
Mr. W. P. Barba ..	0.041	0.015	0.095	0.091	0.041
Mr. A. A. Blair ..	0.040	0.016	0.098	0.091	0.041
Dr. T. M. Drown ..	0.042	0.016	0.104	0.090	0.042
Dr. C. B. Dudley ..	0.040	0.016	0.099	0.097	0.039
Mr. P. W. Shimer ..	0.041	0.017	0.098	0.096	0.039

In explanation of the results, we quote from the Report of the Sub-committee:—

"Sample No. 1 is an ordinary open-hearth steel. Sample No. 2 is a crucible steel. Sample No. 3 is an open-hearth steel to which metallic arsenic was added while in the molten condition in a crucible. Sample No. 4 is an ordinary Bessemer rail steel. Sample No. 5 is the No. 5 sample of the Committee on International Standards, and is an open-hearth steel.

"It will be observed that the agreement in the results on phosphorus obtained by the different chemists is very good. The exceptions are the No. 3 steel, which contains arsenic in considerable amount, and where the discrepancy is 0.009 per cent, and in the No. 4 steel, where the discrepancy is 0.007 per cent. Considerable work was done on the No. 4 sample, in an effort to reconcile discrepancies, and it was found that the turnings from this sample were irregular, and that two different bottles of the sample gave different results. The average of six determinations from one bottle was 0.1057, and the average of five determinations from another bottle was 0.0964 per cent. Furthermore, siftings from quite an amount of the turnings gave 0.140 per cent."

But these methods are long and laborious. It would be impossible with the most rapid of them to get a result in much less than a day, while two days would certainly be required for some of the others. Accordingly, since the demand for rapid phosphorus determinations during the last ten or fifteen years has been very great, an enormous amount of work has been done in trying to meet this demand. Modification after modification has been introduced, and paper after paper has been published on the subject. It is perhaps not too much to say that few chemical journals that publish any original work at all have escaped three or four articles per year, on the determination of phosphorus in iron and steel, or on some phase of a rapid method for such determination. The result of all this work has apparently been constantly in-

* Presidential Address delivered at the Troy Meeting of the American Chemical Society, December 29, 1896. From the *Journal of the American Chemical Society*, xix., No. 2.

creased rapidly, with constantly greater approximations to accuracy. The present state of the matter is perhaps best shown by Thackray (*Trans. Am. Inst. Min. Eng.*, xxv., 370), in his paper "A Comparison of Recent Phosphorus Determination in Steel." This writer sent to some twenty-three different chemists borings from two different samples of steel, with a request to have the phosphorus determined in each sample, and a description of the method used sent with the results. Each chemist was told that samples had been sent to others, but no attempt was made to have any special method used. The chemists embraced a professor in a technical school, the chemist of a large consumer, a number of commercial chemists, and a number of chemists employed by steel and iron works. On one sample thirty-six different results were sent in, and on the other thirty-eight. Twenty-seven different methods were employed, some of the chemists sending in results by two, and even three methods, and some sending duplicate determinations. The results obtained were obtained as follows, the figures being percentages of phosphorus in the steels:—

Sample.	1.	2.
Average of all determinations	0.0496	0.0835
Highest result.. .. .	0.055	0.091
Lowest result	0.045	0.076
Maximum difference	0.010	0.015

The methods employed may be divided on the basis of time required into three classes:—

1st. Those which may be called rapid, and which give a result in two hours or less.

2nd. Those which may be called slow, and which require considerably more than two hours, but still give a result the same day.

3rd. Those which may be called very slow, and which do not give a result until the second day or later.

Thirteen results on each sample were given by "rapid" methods, eleven on the No. 1 sample, and twelve on the No. 2 sample by "slow" methods, and twelve on the No. 1 and thirteen on the No. 2 by "very slow" methods. Arranging the results in accordance with this classification of the methods (and we have some very interesting data), the figures being as before, the percentages of phosphorus in the two steels are:—

Rapid methods.		Slow methods.		Very slow.	
1.	2.	1.	2.	1.	2.
Average ..	0.0499 0.0840	0.0490 0.0826	0.0496 0.0837		
Highest ..	0.054 0.091	0.052 0.086	0.055 0.089		
Lowest ..	0.045 0.078	0.046 0.076	0.046 0.078		
Max. diff. .	0.009 0.013	0.006 0.010	0.009 0.011		

To our minds these figures are very impressive. It is worthy of note—

1st. That the average results given by the "rapid" methods only differ on either steel from the averages given by the "slow" or "very slow" methods, by a little over 0.001 of a per cent.

2nd. That the maximum difference between the highest and lowest results given by the "rapid" methods on either steel is but a trifle greater than is shown by the "slow" or "very slow" methods.

In other words, if we interpret these results correctly they show that the rapid methods for determining phosphorus in steel now known and in use in many laboratories give results that are well nigh as accurate and reliable as those yielded by the longer and more laborious methods, and it must not be forgotten that although we have placed two hours as the time characterising a rapid method, a number of the results given above were obtained by the use of methods which give a single determination in forty-five minutes, and enable one operator to make twenty phosphorus determinations in a day. We are frank to say we do not believe such a showing would have been possible five years ago.

But these results still leave something to be desired. The discrepancy between the highest and the lowest result is still too great. It is, perhaps, a little hazardous to place limits, but we do not think the chemists of the country should be satisfied until they are in possession of a method or methods which are so carefully worked out and so well described that in the hands of different chemists of good fair ability and experience, results will be obtained by all, when working on the same steel, that will not differ from each other more than 0.003 per cent. The Sub-committee on Methods of the International Committee on Standards for the Analysis of Iron and Steel, before referred to, have had in hand now for some two years studies on a rapid and accurate method for the determination of phosphorus in steel. It has been the hope of the Sub-committee that the ideal above given would be attainable by this method. In reality, the work of the Sub-committee has embraced an examination of almost every chemical point involved, taking very little if anything for granted, and checking and proving every step. The work is not yet quite ready for publication, one or two points remaining which are not entirely settled, and it has been deemed advisable to withhold the method until these are completely cleared up.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, May 20th, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

MESSRS. H. E. Gardner, William Barlow, and Paul Thomas White were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Walter Harry Barlow, 152, Osbaldiston Road, Stoke Newington, N.; Ernest Stuart Cameron, 51, Pembroke Road, Dublin; Medwin Caspar Clutterbuck, B.Sc., Ph.D., 61, Beaconsfield Villas, Brighton; Frank William Harbord, Egham; B. J. Harrington, Ph.D., McGill College, Montreal; A. G. Kidston Hunter, Princes Street, Dunedin, N.Z.; John Edwin Mackenzie, B.Sc., Ph.D., 7, Ramsay Garden, Edinburgh; Lionel Walter Kennedy Scargill, B.A., 14, Brunswick Place, W. Brighton; James Porter Shenton, 34, Lansdowne Road, W. Didsbury, Manchester.

Of the following papers those marked * were read:—

*60. "*The Theory of Osmotic Pressure and the Hypothesis of Electrolytic Dissociation.*" By HOLLAND CROMPTON.

The author applies the results obtained by Guye, Ramsay, and Shields, and others in their investigations on the molecular complexity of liquids to the theory of osmotic pressure. It is found that van 't Hoff's view, that the osmotic pressure of the dissolved substance is in dilute solution equal to the pressure which the substance would exercise in the same volume if in the gaseous state, is applicable when both the dissolved substance and the solvent form normal or monomolecular liquids. It may also apply if both liquids are associated. But if the dissolved substance is associated and the solvent is monomolecular, the osmotic pressure is then smaller than the theoretical, and becomes inversely proportional to the factor of association α_1 of the dissolved substance. If the solvent is associated and the dissolved compound is monomolecular, the osmotic pressure is greater than the theoretical, and is directly proportional to the factor of association α of the solvent. If the solvent has also an abnormal vapour density, the factor of association of the

vapour being α , the osmotic pressure is directly proportional to α/λ .

By application of the above conclusions, it is shown that the latent heat of fusion r , melting-point on the absolute scale T , and density at the melting-point, d , of a liquid are connected by the expression $rd/T = \text{const.}$ in the case of monomolecular liquids, or $rdx/T\alpha = \text{const.}$ in the case of associated liquids. The mean value of the constant is 0.099, or roughly 0.1. This formula is exactly similar to the Trouton formula, which connects the latent heat of vaporisation, gaseous density, and boiling-point on the absolute scale of liquids.

The molecular reduction of the freezing-point for monomolecular substances in monomolecular solvents is given by van't Hoff's formula, $E = 0.01976 T^2/r$, or by the derived formula $E = 0.2 Td$. If, however, the dissolved substance or the solvent are associated, this formula no longer applies, but $E = 0.01976 T^2 x/r\alpha x_1$, or $E = 0.2 Td/x_1$. Exceptions to van't Hoff's formula for the molecular reduction of the freezing-point appear, therefore, whenever association of either the dissolved substance or the solvent takes place, and it is shown that those exceptions observed in the case of electrolytes in aqueous solution are in perfect keeping with the view that electrolytes are monomolecular compounds in solution in an associated solvent, e.g., water. The hypothesis of electrolytic dissociation is not only unnecessary in explanation of these exceptions, but is inconsistent with what is now known of the molecular character of liquids.

A connection is supposed to exist between the specific inductive capacity of a liquid and its power of promoting electrolytic dissociation. The author shows that it is only associated liquids that have high specific inductive capacities, and that the specific inductive capacity is approximately proportional to the cube of the factor of association of a liquid. It is therefore not on the degree of electrolytic dissociation of the dissolved substance, but on the degree of association of the solvent, that the conductivity depends, and the view is taken that electrolytes are salts in the monomolecular fluid state in solution in associated solvents.

*61. "Molecular Rotations of Optically Active Salts." By HOLLAND CROMPTON.

A fact which is usually quoted as strong evidence in favour of the hypothesis of electrolytic dissociation, is that salts which contain a common optically active ion—either positive or negative—exhibit, in sufficiently dilute aqueous solution, the same equivalent rotatory power. If, however, electrolytes are salts in the monomolecular fluid condition (preceding paper), the observed regularities indicate that monomolecular salts which contain a common optically active radicle have the same equivalent rotation. Those peculiarities which have been observed in the case of the equivalent rotations of optically active electrolytes in aqueous solution, are shown by the author to be also exhibited by the amylac salts of certain organic acids, when these are examined in the free state and not in solution in any solvent. As electrolytic dissociation is in this case entirely out of the question, the hypothesis becomes an unnecessary one in other instances, and the behaviour of optically active electrolytes is merely in keeping with that of other optically active monomolecular salts.

*62. "Heats of Neutralisation of Acids and Bases in Dilute Aqueous Solution." By HOLLAND CROMPTON.

The constancy of the heat of neutralisation of an acid by a base is usually explained in accordance with the electrolytic dissociation hypothesis by the assumption that the acid, base, and the resulting salt are all in a dissociated state, and that the only change occurring in the system is the formation of water from its ions. In this paper, the author calls attention to the fact that from Thomsen's "Thermochemische Untersuchungen." Band IV., it may be inferred that the replacement in any monomolecular organic compound RH of the H atom by one

and the same radicle M, is attended with a constant heat change, which is independent of the character of R, and that for monomolecular compounds the heat of the reaction $RH - H + M$ is constant if M is constant and independent of variations in R. From this it also follows that the heat of the reaction $ROH - OH + M$ is constant. In the neutralisation of an acid RH by a base MOH, we have the changes $M - OH$, $R - H$, $M + R$, $H + OH$. If M is kept constant then two terms in the reaction will be constant, $M - OH$ and $H + OH$. The only variation is then in $R - H$ and $M + R$. But as shown above, for monomolecular compounds $RH - H + M$ is attended with a heat change that is independent of R, and hence if acids and bases in dilute aqueous solution are monomolecular compounds, the heat of neutralisation of any acid by one and the same base is a constant quantity. It may be shown in similar manner that the heat of neutralisation of any base by one and the same acid is constant, and hence the heats of neutralisation of acids by bases are always the same. The hypothesis of electrolytic dissociation is unnecessary in explanation of the observed phenomena, if it be granted that the dissolved electrolytes are monomolecular compounds.

In the above, since OH is simply another negative radicle R, the heat of the reactions $M - OH$ and $H + OH$ might be expected to exactly balance that of the reactions $H - R$ and $M + R$. This is probably the case when the reactions do not occur in dilute aqueous solution. But in solution, while the acid, base, and salt are in a condition comparable with that of their vapours, the water which is formed in the reaction must be transformed from that state to the liquid state of the solvent by which it is surrounded. This implies that the heat of neutralisation of an equivalent of an acid by an equivalent of a base in aqueous solution contains as main factor the heat of condensation of a molecule of water. This latter quality has a value of about 10,800 cal., and the mean value of the heat of neutralisation is 13,500 cal. The difference between the two values is to be mainly attributed to the state of partial association of the base.

DISCUSSION.

Mr. PICKERING said whether Mr. Crompton had established his views or not, he had succeeded in throwing much new light on the subject under examination, and had given us further evidence that the theory of dissociation was not the only one through which we might look for an explanation of the phenomena of dissolution.

By way of criticism, the speaker suggested that the means of recognising a liquid to be of the associated or non-associated class at the freezing temperatures was somewhat imperfect, and might, in many cases, lead to erroneous conclusions. He doubted, also, whether the numbers obtained showed that the same solvent indicated consistently the same degree of association when pitted against various monomolecular solutes, as should be the case, and whether the same associated solute, when pitted against various monomolecular solvents, gave similarly consistent results. A stronger objection, however, might be raised in the behaviour of diatomic and triatomic electrolytes in water. According to Mr. Crompton's views, these should both give values of 55.2 for the depression of the freezing-point when in extreme dilution; the triatomic electrolytes do so, but diatomic electrolytes give values which show little or no tendency to surpass 37, which is only double instead of three times the "normal" value.

As regards the heat of neutralisation, the speaker considered Mr. Crompton's application of a general principle which has been established in organic transformations to similar transformations in inorganic solutions to be both legitimate and ingenious. The simplicity of the principle for organic substances, no doubt, depends on the fact that these substances are nearly saturated compounds, and in dilute solutions of inorganic compounds we are probably also dealing with saturated compounds. Some years ago, the speaker brought before the Society an explanation of

the constancy of the heat of neutralisation which was based on chemical grounds, without recourse to the theory of dissociation. Residual affinity was the explanation which was offered, and Mr. Crompton's explanation could be improved by taking residual affinity into consideration. Mr. Crompton accounts for the heat evolved on neutralisation by the condensation of the molecule of water formed; this condensation should certainly be recognised (a fact which the speaker had overlooked in his own communication on the subject), but the heat evolved by it falls short of that of neutralisation by some 3000 cal., and it seems probable that this excess may be accounted for by the salt formed becoming, in the presence of water, more fully saturated than either the acid or the alkali. Each of these latter contains a radicle, H and OH, which is identical with one of the radicles in water itself, and such compounds would, therefore, probably not have their residual affinity entirely saturated by the water, whereas this is not so with the salt, and there is nothing in its case to prevent complete saturation.

Mr. W. C. D. WHETHAM said that although it was impossible to criticise such an interesting paper without having considered its details, he would like to ask Mr. Crompton how he would explain the phenomena of electrical conductivity. On the theory that the ions were free from each other, the observed fact that the conductivity of a dilute solution varied as the concentration was at once explained. The alternative supposition, that the ions worked their way through the solution by means of a continual series of interchanges between the opposite parts of molecules at the instants of collision, would lead to a different result, for the frequency with which such collisions would occur, and therefore the ionic velocities must vary as the square of the concentration, and since the conductivity depended on the product of the number of ions and their average velocity, it would be proportional to the cube of the concentration.

Then, again, the fact that the velocity of an ion in dilute solution was independent of the other ion present, not only as calculated from the conductivity, but also as directly observed, seemed to favour the idea of dissociation, and was of greater weight than other additive relations, since it involved the properties of the ions when in motion.

The successful calculation of potential differences at the contact of two solutions on the assumption that the faster-travelling ion moved independently of the other, and so diffused more quickly, must also be remembered. Such phenomena as these must be explained before the dissociation theory could be abandoned. No doubt the theory presented many difficulties, and a successful attempt to explain the facts in some other way would be of extreme interest; but at present the evidence in favour of the dissociation theory seemed very strong.

Dr. SHIELDS, after referring to the difficulty of discussing the paper until all the details were before them, stated that he was not satisfied that Mr. Crompton had made out his case that abnormally large osmotic pressures were due to the association of the solvent. According to the well-known equation, the osmotic pressure, π , of a solution containing n molecules of dissolved substance in N molecules of solvent is represented thus:—

$$\pi = \frac{n}{N} \cdot \frac{RT \ 1000 \rho}{M};$$

where M denotes the molecular weight of the solvent, ρ the specific gravity of the solution, T the absolute temperature, and R is a constant, viz., 0.0819 litre-atmospheres, when we express the osmotic pressure in atmospheres, and the volume of the solution containing 1 g.-molecule in litres. In the above equation, the product NM is the weight in grms. of the solution containing n g.-mols. of the dissolved substances.

If we make up a dilute solution to contain, by intention, n g.-mols. of dissolved substance in N g.-mols. of a solvent

supposed, in the first instance, to be normal or monomolecular, then we get a certain definite value for the osmotic pressure. If, however, the solvent is associated, and x is a measure of its molecular complexity, then instead of having weighed out N g.-mols. of solvent, we have in reality only N/x , and since the weight of the solution remains the same, the osmotic pressure must be—

$$\pi = \frac{n}{N/x} \cdot \frac{RT \ 1000 \rho}{xM};$$

or, in other words, remain uninfluenced by the degree of association of the solvent.

As regards aqueous salt solutions, Dr. Shields thought Mr. Crompton would encounter serious difficulties in attempting to explain why dilute solutions of binary compounds, such as potassium chloride had a maximum osmotic pressure of twice the theoretical value, whilst compounds like calcium chloride gave three times the pressure one would expect.

Dr. Shields also called attention to the fact that associated liquids, such as water, become less associated as the temperature is raised, and asked whether when the particular temperature were reached at which water becomes "normal," salt solutions also become "normal," i.e., show the theoretical osmotic pressure corresponding to that temperature and otherwise behave like indifferent substances or non-electrolytes.

Mr. CROMPTON, in reply, explained that in assigning to a particular liquid a monomolecular or an associated character the general results of the work of Guye, Ramsay and Shields, and others had as far as possible been adhered to. That the molecular reduction of the freezing point of water by electrolytes was in certain cases, even in the most dilute solutions, below the value required for monomolecular compounds, indicated that the salt was originally associated and that the complex molecules only broke down slowly with rising dilution. Similar instances could be observed in the case of solutions of associated compounds in other solvents, e.g., benzene. Alcohol, which in concentrated solution in benzene gave a molecular weight far higher than the normal, would be found to give correct values in very dilute solution. On the other hand, acetic acid gave even in very dilute solution in benzene a molecular weight of about 110 in place of 60, the splitting up of the associated molecules taking place apparently with greater difficulty in the case of this compound than in that of alcohol. The adequacy of the dissociation hypothesis to explain the electrical properties of salt solutions had not been called in question, but it had been shown that the hypothesis gave no true account of certain other properties of salt solutions which it had hitherto professed to explain. The additive character of the molecular conductivities of dilute salt solutions was merely in keeping with the additive character of nearly all the properties of monomolecular compounds in the fluid condition, as, for example, the molecular volumes, the molecular refractions, the molecular viscosities. If a dissociation hypothesis were adopted to explain additive properties in one case, this would have to be extended to all, and such a thing as a monomolecular fluid compound would be non-existent.

*63. "A Comparative Crystallographical Study of the Normal Selenates of Potassium, Rubidium, and Cesium." By A. E. TUTTON.

The main conclusions of this investigation, which is analogous to the one formerly presented concerning the corresponding sulphates (*Trans.*, 1894, lxx., 628), are as follows.

1. The order of solubility of the three salts follows that of the atomic weights of the three respective metals contained.

2. The values of the morphological angles of the crystals of rubidium selenate are without exception intermediate between those of the analogous angles of the potassium and cesium salts. The angles are therefore a function of the atomic weight of the metal present.

3. The morphological axial ratios of rubidium selenate are likewise intermediate.

4. The usual habits of the crystals of the three salts exhibit a progressive development of the primary forms, following the progressive change in atomic weight.

5. The directions of cleavage are identical.

6. The relative density and molecular volume increase when a lighter is replaced by a heavier alkali metal. The increase in density is greater when potassium is replaced by rubidium than when the latter is replaced by caesium, and the increase in molecular volume is, on the contrary, greater when rubidium is replaced by caesium. The replacement of sulphur in the sulphates by selenium is accompanied by an increase of molecular volume varying from 6.5 to 6.7 inversely as the weight of the initial molecule.

7. The replacement of potassium by rubidium, and of the latter by caesium, is accompanied in each case by an increase in the separation of the centres of contiguous units of the homogeneous crystal structure, along the directions of each of the morphological axes, the influence of the nature of the alkali metal becoming relatively greater as the atomic weight rises. An extension of volume in all directions also accompanies the replacement of sulphur by selenium.

8. An increase of refractive index is observed to accompany an increase in the atomic weight of the alkali metal, and the increase becomes relatively greater as the atomic weight rises. The replacement of sulphur by selenium is also accompanied by an increase of refractive index, and such increase diminishes in amount as the weight of the initial molecule increases.

9. If the closed optical ellipsoidal figures, the optical indicatrices, of the three salts were constructed about the same origin, the indicatrix of the caesium salt would contain within it that of the rubidium salt, and this again would contain that of the potassium salt. The indicatrix of the rubidium salt would lie nearer to that corresponding to the potassium salt.

10. The replacement of one alkali metal by another of higher atomic weight is accompanied by a diminution of the already feeble double refraction. In the convergence of the axial values of the optical indicatrix towards unity the c value proceeds much more rapidly than the others.

11. The latter fact causes a reversion of the sign of double refraction from positive to negative on attaining the caesium salt.

12. The optic axial angles are precisely such as would naturally follow from the progressive development of the optical indicatrix; a change of direction of the acute bisectrix and of the optic axial plane occurs when the caesium salt is reached, as the direct result of the continuity of the progression according to atomic weight.

13. The optical properties of the selenates exhibit marked specific differences from those of the sulphates, owing to the progressively different effect of replacing sulphur by selenium in the three sulphates, but the whole of the relationships of these optical properties exhibited by the three salts of each group are of a precisely parallel nature, being functions in each case of the atomic weight of the alkali metal which they contain.

14. Progressive changes occur in the optical properties on raising the temperature, following, even to the least detail, the order of the atomic weights. An interesting direct consequence is that a 60° prism of caesium selenate whose vibration-directions are parallel to b and c affords at 90° C. only one image of the spectrometer slit, the two images usually observed coinciding at this temperature, the crystal being then apparently uniaxial.

15. A further consequence of the foregoing is that the crystals of caesium selenate exhibit unique interference phenomena in convergent polarised light when their temperature is raised, including crossed axial plane dispersion, and two reversals of the sign of double refraction. Section-plates perpendicular to all three axes in turn re-

quire to be employed in order to follow the optic axial changes even as far as 280° C.

16. The whole of the molecular optical constants of rubidium selenate are intermediate between those of potassium and caesium selenates. The replacement of sulphur by selenium is accompanied by an increase of molecular refraction of 3.4–3.8 Lorenz or 6.2–6.7 Gladstone units, according to the direction chosen for comparison. The relations of the three salts of each group as regards molecular refraction are identical, but the actual differences are slightly greater in the selenate group than in the sulphate group.

17. The molecular refraction of each of the three selenates for the state of solution in water is approximately the same as the mean of the three values for the crystal. When potassium selenate is dissolved in water, its refraction equivalent rises by 2.8 per cent; in the case of rubidium sulphate, a less rise of 1.0 per cent is observed, while for caesium selenate there is no longer a rise but a decrease, to the extent of 0.5 per cent. These slight differences, due to change of state, thus exhibit a progression varying directly as the specific refractive energy and inversely as the atomic weight of the alkali metal contained in the salt. After subjecting Kanonnikoff's value for dissolved potassium sulphate of revision, precisely similar differences for the two states are shown to exist in the sulphate group.

18. The author finally concludes as regards the selenates that—

The whole of the morphological and physical properties of the crystals of the rhombic normal selenates of potassium, rubidium, and caesium are functions of the atomic weight of the alkali metal present.

19. It is shown that the joint results of the investigations of the sulphates and selenates agree with the assumption that—

The characters of the crystals of isomorphous series are functions of the atomic weight of the interchangeable elements, belonging to the same family group, which give rise to the series.

DISCUSSION.

Dr. GLADSTONE remarked that everyone recognised in a general way that in groups of analogous elements there is a gradual progression in the properties, the middle member of the group being intermediate, not only in atomic weight, but also in other respects. The value of Mr. Tutton's elaborate papers, is, that he has proved this up to the hilt quantitatively in the case of two similar, well defined groups of salts, and that with regard to a large number of properties. The change in the specific refraction of the selenates of the alkalis in their crystalline and their dissolved condition is especially instructive, as it involves the change from *plus* in potassium and rubidium to *minus* in caesium. The correction of Kanonnikoff's number for the potassium sulphate which Mr. Tutton has made brings the atomic refraction back to a figure practically identical with that published in Dr. Gladstone's paper of 1870, viz., 33.11.

*64. "The Platinum-Silver Alloys; their Solubility in Nitric Acid." By JOHN SPILLER.

Referring to the published statements in the text-books, and particularly to those in Percy's "Metallurgy" and Bloxam's "Chemistry," according to which 5 or even 9 per cent of platinum followed the silver into solution when their alloys were treated with nitric acid, the author investigated the properties of ten graduated alloys constituted as follows:—Series I, containing 12, 9 and 5 per cent of platinum; series II, containing 2, 1.5, 1 and 0.75 per cent of platinum; series III, containing 0.5, 0.4 and 0.25 per cent of platinum. These alloys were prepared by fusion of the requisite proportions of silver and platinum under a gas-air blow-pipe flame in shallow porcelain cups, and then attacked by nitric acid of three different strengths, when it was found that the ordinary

concentrated acid of 1.42 sp. gr., warmed, proved the best solvent, but that even under the most favourable conditions no more than 0.75 to 1.25, mean 1 per cent of platinum, could be dissolved along with the silver.

When diluted nitric acid of 1.2 sp. gr. was employed, the maximum amount of platinum taken up was only about 0.25 per cent; whilst the highly concentrated acid of 1.50 sp. gr. proved altogether inappropriate, giving a bulky, insoluble product consisting of platinum black, intermixed with nearly the whole of the silver nitrate formed.

It would appear, then, that Berthier's account, quoted by Percy, and the statement in Bloxam's "Chemistry" are incorrect.

DISCUSSION.

Mr. VERNON HARCOURT suggested that the composition of the alloys of platinum and silver might vary with the temperature at which they were formed, and that Mr. Spiller should determine the solubility of alloys formed at higher temperatures than those he had employed.

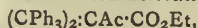
Mr. FRISWELL thought that impurities in the nitric acid might account for some of the discrepant statements on record.

65. "Dalton's Law in Solutions. The Molecular Depression of Mixtures of Nonelectrolytes." By MEYER WILDERMAN, Ph.D.

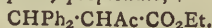
Since van 't Hoff has shown that the generalisations arrived at by Boyle and Guy-Lussac in the cases of gases are equally applicable to dissolved substances in dilute solutions, the conclusion must be drawn that the third gaseous law, the law of Dalton, holds for dilute solutions also, this being a necessary consequence of the nature of osmotic pressure. Following up the thermodynamic considerations of Planck, the equations for mixtures of two or more electrolytes and the experimental proof of them are given.

66. "The Action of Bromdiphenylmethane on Ethyl Sodacetoacetate." By G. G. HENDERSON, D.Sc., M.A., and M. A. PARKER, B.Sc.

While bromtriphenylmethane and ethyl sodacetoacetate interact to give a disubstituted derivative—



and ethyl acetoacetate, bromdiphenylmethane, on the other hand, appears to yield only a monosubstituted ester, ethyl α -acetyl- β -diphenylpropionate,—



This substance was prepared by heating bromdiphenylmethane (1 mol.) and ethyl sodacetoacetate (1 mol.) in presence of pure dry benzene or xylene till the reaction was completed, filtering from sodium bromide, concentrating the benzene solution, and purifying the crystals, which then separated, by recrystallisation from alcohol. It crystallises in shining, colourless needles, m. p. 85°, is sparingly soluble in alcohol but readily in benzene, and decomposes almost entirely when distilled.

On hydrolysis of this ester with cold dilute aqueous potash, a small quantity of α -acetyl- β -diphenylpropionic acid, $\text{CHPh}_2\text{CH}(\text{Ac})\text{COOH}$, was obtained in the form of extremely unstable crystals, which melt about 90° and decompose at a slightly higher temperature. The salts of this acid are also very unstable. β -diphenylethylmethylketone, $\text{CHPh}_2\text{CH}_2\text{CO}\cdot\text{CH}_3$, was prepared by hydrolysing the ester with hot dilute alcoholic potash. It crystallises in colourless prisms which melt at 87.5° and distils with almost no decomposition at 315°. It is fairly readily soluble in alcohol, and very readily in benzene. The oxime, $\text{CHPh}_2\text{CH}_2\text{C}(\text{CH}_3)\text{N}\cdot\text{OH}$, forms small, colourless crystals, m. p. 86–87°. It is sparingly soluble in alcohol but readily soluble in benzene. The semicarbazone, $\text{CHPh}_2\text{CH}_2\text{C}(\text{CH}_3)\text{N}\cdot\text{NH}\cdot\text{CO}\cdot\text{NH}_2$, crystallises from alcohol in small, white clusters of minute crystals, which melt at 181°. It is sparingly soluble in alcohol and in benzene.

PHYSICAL SOCIETY.

Ordinary Meeting, May 28th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

Mr. ELDER read a paper communicated by Dr. ALBERT A. GRAY, M.D., on "The Perception of the Difference of Phase by the Two Ears."

The investigation relates to certain acoustical results obtained some years ago by Dr. S. P. Thompson; they may be summarised as follows:—(a). When two simple tones in opposite phases are conveyed separately, through tubes or otherwise, to the two ears, the sensation of sound appears localised at the back of the head. (b). If the respective tones from two forks mistuned to give "beats" are conducted separately to the two ears, they still produce the sensation of "beats"; and, to the observer, this sensation also seems localised at the back of the head. The "beats" are distinct, but there are no true silences,—at any rate so long as attention is fixed on the note. (c). Although "beats" are heard under these circumstances, no beat-tones are discernible by the binaural method. The author proceeds to explain the phenomena on the assumption that there is a physiological connection between the nerves of both ears. His evidence is derived from the following experiments:—(d). A vibrating fork is held opposite one ear; the opposite ear is then closed by a finger; the sound of the fork now appears louder to the open ear. (e). If the fork is held opposite one ear, and the chain of ossicles of the second ear is then pressed gently inwards by a fine probe, the sound of the fork is heard with increased loudness by the first ear. (f). If the chain of ossicles in the second ear is dragged outwards by rarefaction of the air in the meatus, the above changes in loudness are no longer perceptible. The theory put forward by the author in explanation of these results is, that they are due to reflex contractions of the tensor tympani or stapedius (or more probably both) of the first ear. A further observation, of Pollak, is also brought to bear upon the question, *i.e.*, (g). Stimulation of one cochlea by sound causes contraction of the tensor tympani of both ears, and the contraction is permanent while the sound continues. This is known to be true for the lower animals, and is probably true for man.

With regard to (a), the author observes that the muscular sense is there being appealed to in a manner quite new to it. The tympani are by nature trained each to relax or expand with the other, and they are thrown out of reckoning if the phases differ. Or again, the stimuli from the two ears may collide at one of the lower nerve centres, and thus be annulled before any intimation has been received by the brain. The path taken by such stimuli is from the nucleus of one nerve, just after its entrance into the medulla, across to the corresponding nucleus of the opposite side. In these nuclei the stimuli from both ears mix. Some of the nerve-fibres have no nuclear intercommunication at the base of the brain; consequently, stimuli passing by these paths are not subject to interference; this agrees with (b), where the silences are not complete. (h). It is to be observed that beat-tones are sometimes perceived by the ear under circumstances where they cannot set a resonator into vibration. This indicates that beat-tones may be produced either in the ear or nerve-centres of the listener, and not exteriorly. (i). It has been shown by Dr. Thompson that when two simple tones, such as in ordinary hearing produce a differential tone, are led singly to the ears, no differential tone is heard. From this the author concludes that differential tones are not produced in the mind of the listener, nor in any of the cerebral centres. From (h) and (i) together, the point of production is restricted down to the ear itself; something of the sort was suggested by Helmholtz. Again, from (g), it appears that when two notes are sounded so as to give a differential tone, the tensor tympani must be in a state of continual contraction,

for the intervals of silence are too short to permit of any relaxation. Meanwhile, there are certain periods during which the tympani membranes are not acted upon by any force external to the ear. The author is of opinion that if the movements of the ossicles upon one another were absolutely frictionless, the membranes would come to rest in a position where the force of the contracting muscle was balanced simply by the tension of the membrane and the ligaments of the ossicles; but since the articulations of the ossicles have some friction, the equilibrium is otherwise, and he conjectures that the state of affairs is such that any force acting upon the hammer, tending to draw it inwards, produces a slight jerk, and this repeated gives the necessary impulses for the sensation of differential tones. The mechanics of this theory is not fully worked out.

Mr. J. ROSE-INNES read a paper on "*The Isothermals of Isopentane.*"

The author takes advantage of the recent experimental work of Ramsay and Young, upon the thermal properties of isopentane, to test a formula giving the relation of pressure to temperature for gases generally, over a considerable range of volume. From the linear equation, $p = bT - a$, for the pressure at constant volume, where a and b are functions of the volume, no formula could be found to give close agreement with observed results. More definite results are obtained by examining a quantity depending upon a and b together; such a quantity is the temperature τ , at which, for each volume, the substance behaves as a perfect gas. It is shown by tables that τ is nearly a constant for volumes from 350 to about 8. Below vol. 8 it diminishes very rapidly with volume. A further investigation refers to the—

$$\left[(av_2)^{-1}; v^{-\frac{1}{2}} \right]$$

curves of Young, for isopentane, and a corresponding formula. At vol. 3.4 on this curve there is a decided peak, suggesting discontinuity. Ether gives a similar curve, and the question arises whether such curves would not be better represented by two or more equations.

Prof. YOUNG said the diagrams representing the observed and calculated isothermals were probably the best ever obtained. Divergence among the values of τ was explained in part by the smallness of the angle between the theoretical isochor for a perfect gas, and the real isochor. The point of coincidence was difficult to define. Moreover, the values of τ were obtained from "unsmoothed" values of v . The evidence against the linear law consisted in a certain similarity in the shape of the different curves. It was not easy to see where experimental errors could come in. The peak was a very striking feature of the curves, and the agreement between the results with ether and those of isopentane was very remarkable. These two substances had their boiling-points close together, their critical temperatures close together, and their molecular weights nearly alike. The two substances not only agreed in each giving a peaked curve, but the peak corresponded to almost identical volumes. Prof. Young hoped at some future time to examine normal pentane, and to determine whether τ was a constant for this substance also.

The PRESIDENT proposed votes of thanks to the authors of the papers, and the meeting was adjourned until June 11th.

Results obtained by the Transformation of Ammonium Carbonates into Urea.—Leon Bourgeois.—The author finds that 6.0 per cent of the carbonate employed is represented by the average amount of urea obtained, the figures varying from 3.2 to 9.5 per cent when using commercial sesquicarbonate: bicarbonate of ammonia gave 2.5 to 2.9 per cent of urea; and carbamate of ammonium, $\text{CONH}_2\text{ONH}_4$, gave 2.6 to 3.7 per cent of urea.—*Bulletin de la Société Chimique de Paris*, xii.-xiii., No. 9.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Journal de Pharmacie et Chimie.

Series 6, vol v., No. 10.

Contribution to the Study of Pilocarpine and Pilocarpidine.—A. Petit and M. Polonovski.—To show the difference existing between pilocarpine and pilocarpidine a table has been compiled giving the properties and principal characteristics of these two bases and their derivatives. From an intimate knowledge of these properties the authors are enabled to state that the impurity often found in commercial salts of pilocarpine is nothing else but pilocarpidine. These are, as is well known, the alkaloids of *jaborandi*, and the authors have proved that pilocarpidine exists in the plant itself. This assertion is based on the following facts:—1. In the absence of strong acids and alkalis, the action of boiling water alone will not explain the formation of the large quantities of pilocarpidine which is sometimes obtained, in view of its feeble action on pilocarpine. 2. Even when carefully guarding against heat during its preparation we always find in the end more or less considerable quantities of pilocarpidine. 3. The return of pilocarpidine varies considerably in quantity, viz., from 5 per cent up to 75 per cent, according to the species of *jaborandi* used. And, 4. That the stalks generally produce a much greater quantity than the leaves of the same plant.

Revue Générale des Sciences Pures et Appliquées.

No. 9.

Production and Utilisation of Acetylene.—F. Dommer.—Lighting by means of acetylene gas has recently experienced a temporary check, attributable to the absence of carbide of calcium on the market, and to the fact that there have been a few accidents which were caused by inexperience and faulty apparatus. Acetylene gas when properly dealt with is no more dangerous than coal-gas, provided it is not subjected to greater pressure than two atmospheres. Still the manufacture of carbide of calcium may now be regarded as a definite industry, and in this paper the author goes fully into the merits and disadvantages of the different furnaces used for its manufacture, all based, it is needless to say, on M. Moissan's electric furnace; but besides the chemical aspect of the question, M. Dommer thoroughly considers and analyses the question of cost, of fitting and erecting a factory, and running it successfully when completed.

The New Tuberculine of Koch.—Dr. R. Romme.—Seven or eight weeks ago the medical world was startled by the news that Dr. Robert Koch had discovered a true cure for tuberculosis. A few days afterwards a memoir was published in the *Deutsche medicinische Wochenschrift*, in which Koch described his new tuberculine. It differs entirely from the *old* of 1890, which was admittedly a failure. The latter was a glycerin extract of tuberculous cultures, and when injected subcutaneously caused local inflammation, accompanied by a general reaction, high fever, palpitation, sickness, &c. The new tuberculine, which is obtained by the mechanical trituration of the *Bacillus tuberculosis*, dried by successive "centrifugations" of the matter added to water, causes none of these symptoms—no morbid phenomena appear. Both are toxic, but while the *old* accelerated the disease, and, to use the true word, killed the patient, the *new* kills the disease when not too far advanced, and thus cures the patient. Animals and human beings are immunised by progressive increasing injections until they are quite insensible to tuberculosis. This paper is principally devoted to explaining in detail

the directing idea which guided Dr. Koch in his work and the technical procedure which ensured his success.

Bulletin de la Société Chimique de Paris.
Vol. xvii.-xviii., No. 9.

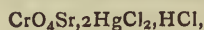
M. Béchamp made a first communication on soluble ferments.

M. Matignon announced the preparation of carbide of sodium and monosodic acetylene, both pure, by the direct action of acetylene on sodium. The reactions of these bodies will be communicated later on.

M. Léger has studied the action of hypobromite of sodium in excess on certain phenols, and gives a short account of his results. He proposes to extend his researches to a certain number of bodies, enclosing one or more molecules of phenolic OH, and to weigh the products formed.

On a Reaction of Carbonic Oxide.—A. Mermet.—Air containing 1/500th to 1/5000th of carbonic oxide will decolourise a weak solution of permanganate of potash acidulated with nitric acid. The action is accelerated by the addition of nitrate of silver, the time varying from 1 hour to 24 hours. The strengths of the solutions used to demonstrate this new reaction are—2 or 3 grms. of nitrate of silver in 1 litre of water; the permanganate of potash is prepared by boiling a litre of distilled water containing a few drops of nitric acid (free from HCl), then adding a strong solution of permanganate drop by drop until the rose colour is persistent; this is to destroy what organic matter may be present. On cooling dissolve 1 grm. of crystallised permanganate of potassium and add 50 c.c. of pure nitric acid, and keep in the dark. To perform the experiment 20 c.c. of the silver solution is mixed with 1 c.c. of the permanganate and 1 c.c. of pure nitric acid, and made up to 50 c.c. with distilled water free from organic matter. On passing air which has been cleansed by first being passed through a tube containing cotton-wool, another containing phosphoric anhydride, another containing baryta water, &c., through this liquid, the decolouration is complete.

Action of Chromate of Strontium on Mercuric Chloride.—H. Imbert and G. Belugou.—The authors find that chromate of strontium and mercuric chloride in a hydrochloric acid solution form a double salt—



which is undecomposable by water. They are also able to predict the existence of a certain number of double salts containing an excess of hydrochloric acid. In a further note on the same subject M. Belugou obtains analytical results which lead him to believe that a basic mercuric chromate is also formed which is mixed with the principal substance as an impurity.

Preparation of $\alpha\alpha\beta$ -Triphenylethane.—J. Rawitzer.

Benzylidenes-diphenyl-hydrazine, and their Derivatives; and the Transformation of these Bodies into Dibenzylidene-diphenyltetrazol.—H. Causse.—Neither of these last two papers are suitable for abstraction.

Essence of Cedar Wood.—L. Rousset.—Absolutely pure essence of cedar wood, free from any adulteration, was obtained from the pencil-works at St. Paul-en-Jarez, and fractionated *in vacuo*. Four-fifths of the essence constitutes a hydrocarbide, boiling at 125° to 130°, at 9 m.m. pressure. This hydrocarbide is *cedzene*, $\text{C}_{15}\text{H}_{24}$. It is a sesquiterpene, and acts on polarised light $\alpha_D = -47^\circ 54'$.

Research on Essence of Geranium.—Eugene Charabot.—The author finds that the *lævo*-rotatory ether, stated to be one of the constituents of essence of *pelargonium*, is not contained in essence of *palmrosa*.

NOTES AND QUERIES.

*** Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Thorium.—Could any reader give outline of cheap and practical method for the extraction of thorium from monazite sand?—F. B.

MEETINGS FOR THE WEEK.

TUESDAY, 8th.—Royal Institution, 3. "The Heart and its Work," by Dr. Ernest H. Starling.

THURSDAY, 10th.—Royal Institution, 3. "Wordsworth and Coleridge," by Churton Collins, M.A.

FRIDAY, 11th.—Royal Institution, 9. "On Diamonds," by William Crookes, F.R.S.

Physical, 5. "The Effect of Sea-water on Induction Telegraphy," by C. S. Whitehead. "A New Definition of Focal Length, and an Instrument for its Determination," by T. H. Blakesley. "Decomposition of Silver Salts under Pressure," by Dr. J. E. Myers and Dr. F. Braun. "New Way of Determining Hysteresis in Straight Strips," by Dr. Fleming, F.R.S.

SATURDAY, 12th.—Royal Institution, 3. "Music in England during the Reign of Queen Victoria," by J. A. Fuller Maitland, M.A.

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THE CHEMICAL NEWS.

VOL. LXXV., No. 1959.

THE LIQUEFACTION OF FLUORINE.*

By H. MOISSAN and J. DEWAR.

THE physical properties of a large number of mineral and organic compounds of fluorine indicated theoretically that the liquefaction of fluorine could only be accomplished at a very low temperature. Whilst the chlorides of boron and silicon are liquid at ordinary temperatures, the fluorides are gaseous and very far from their points of liquefaction. This is also true with the organic compounds; chloride of ethyl boils at $+12^{\circ}$ C., and the fluoride of ethyl at -32° .† Chloride of propyl boils at $+45^{\circ}$, and the fluoride of propyl at -2° .‡

Similar observations have been made by Paterno and Oliveri,§ and by Vallach and Heusler.||

Gladstones' experiments on atomic refraction¶ can well be compared with these facts.

In fact fluorine by certain of its properties resembles oxygen, though at the same time it is distinctly at the head of the chlorine group.

The conclusion to be drawn from these observations appears to be that fluorine can only be liquefied with great difficulty. One of us showed that at a temperature of -95° , at the ordinary pressure, there is no change at all.**

In the new experiments which we now publish, fluorine was prepared by the electrolysis of fluoride of potassium in solution in anhydrous hydrofluoric acid. The fluorine gas was freed from vapours of hydrofluoric acid by being passed through a serpentine of platinum, cooled by a mixture of solid carbonic acid and alcohol. Two platinum tubes filled with perfectly dry fluoride of sodium completed the purification.

The apparatus used for liquefying this gas consisted of a small cylinder of thin glass, to the upper part of which was fused a platinum tube. This latter contained in its axis another smaller tube, likewise of platinum. The gas to be liquefied enters by the annular space, passes through the glass envelope, and escapes through the small inner tube. This apparatus was fused to the tube by which the fluorine was supplied.

In these experiments we used liquid oxygen as the refrigerant. It was prepared according to the method already described by one of us—and this research, we may remark, required several litres.††

The apparatus being cooled down to the temperature of quietly-boiling liquid oxygen (-183°), the current of fluorine gas passed through the glass envelope without becoming liquid. But at this low temperature, it has lost its chemical activity, and no longer attacks the glass.

If we now make a vacuum over the oxygen, we see, as soon as rapid ebullition takes place, a liquid collecting in the glass envelope, while gas no longer escapes from the apparatus. At this moment we stop with the finger the tube by which the gas had been escaping, so as to prevent air from entering, and the glass bulb soon becomes full of a clear yellow liquid, possessed of great mobility. The colour of this liquid is the same as that of fluorine gas when examined in a stratum one metre thick. According to this experiment, fluorine becomes liquid at -185° .

As soon as this little apparatus is removed from the liquid oxygen the temperature rises, and the yellow liquid begins to boil with an abundant disengagement of gas, having all the energetic reactions of fluorine.

We took advantage of these experiments to study some of the reactions of fluorine on bodies kept at extremely low temperatures.

Silicon, borax, carbon, sulphur, phosphorus, and reduced iron, cooled in liquid oxygen and then placed in an atmosphere of fluorine, did not become incandescent. At this low temperature fluorine did not displace iodine from iodides. However, its chemical energy is still sufficiently great to decompose benzene and essence of turpentine with incandescence, as soon as their temperatures rose to -180° . It would thus seem that the powerful affinity of fluorine for hydrogen is the last to disappear.

There is still another experiment we ought to mention. When we pass a current of fluorine gas through liquid oxygen, a flocculent precipitate of a white colour, which quickly settles to the bottom, is rapidly formed. If we shake up this mixture and throw it on a filter, we separate the precipitate, which possesses the curious property of deflagrating with violence as soon as the temperature rises.

We intend to follow up the study of this body, as well as that of the liquefaction and solidification of fluorine, which demand further experiments.—*Comptes Rendus*, vol. cxxiv., No. 22, p. 1202.

ON THE DISTILLATION OF VERY DILUTE MIXTURES OF ETHYLIC ALCOHOL AND WATER.

APPLICATION TO THE ESTIMATION OF ALCOHOLIC SOLUTIONS CONTAINING ONLY 1/3000TH TO 1/10,000TH PART.

By M. NICLOUX and L. BAUDUER.

LAST June one of us brought forward a method of estimating alcohol in solutions containing only 1/500th to 1/3000th part (M. Nicloux, "Dosage de l'Alcool Éthylique dans les Solutions où cet Alcool est Dilué dans des proportions comprises entre 1/500 et 1/3000," *Comptes Rendus de la Soc. de Biol.*, 10th Series, vol. iii., p. 841, July 31, 1896). We will recall this method in a few lines.

"If to a very dilute solution of alcohol (from 1/500th to 1/3000th part) a weak solution (2 per cent) of bichromate of potash be first added, then a little sulphuric acid, the alcohol becomes oxidised and the bichromate becomes a chromic salt, in proportion to the quantity of alcohol present.

"If the bichromate is not in excess the solution is bluish green, the colour of weak sulphate of sesquioxide of chromium. If, on the contrary, it be ever so little in excess, it is yellowish green. The difference between the two tints is very easily noticed.

"By using 5 c.c. of the alcoholic solution under examination and a solution of 20 grms. per litre of pure crystal-

* M. Moissan brought all his apparatus for the production of fluorine to the Royal Institution on the occasion of his lecture there on Friday, the 28th of May. The next day the writer had the good fortune to witness in the laboratories of the Institution by M. Moissan and Professor Dewar some of the experiments which resulted in the liquefaction of fluorine. These experiments mainly owed their success to the unrivalled appliances for the production of intense cold possessed by the Institution, and the skill and experience of Professor Dewar and his assistants in preparing a special apparatus suitable for the examination of, and experimenting with, fluid fluorine, and in the manipulation of large quantities of liquid air.—W. C.

† H. Moissan, "Propriétés et Préparation du Fluorure d'éthyle," *Ann. de Chim. et de Phys.*, Series 6, vol. xix., p. 266.

‡ Meslans, *Comptes Rendus*, vol. cviii., p. 352.

§ Paterno and Oliveri, "Sur les trois Acides Fluobenzoniques Isomères, et sur les Acides Fluotoluidique et Fluonitrique," *Gazzetta Chimica Italiana*, vol. xii., p. 85, and vol. xiii., p. 583.

|| Vallach and Heusler, *Annales de Liebig*, vol. ccxliii., p. 219.

¶ J. H. Gladstone and G. Gladstone, "Refraction and Dispersion of Fluobenzene and Allied Compounds," *Phil. Mag.*, Series 5, vol. xxii., p. 1.

** H. Moissan, "Nouvelles Recherches sur le Fluor," *Ann. de Chim. et de Phys.*, Series 6, vol. xxiv., 224.

†† J. Dewar, "New Researches on Liquid Air," Royal Institution of Great Britain, 1896, and *Proc. Roy. Inst.*, 1893.

lised bichromate of potash, of which 1 c.c. is equal to 1/1000th c.c. of absolute alcohol in the solution used—or, what comes to the same thing, 0.1 per cent in volume, the number, *n*, of cubic centimetres or fractions of cubic centimetres used to obtain the yellowish green colouration, which indicates the slight excess of bichromate, will at once give the amount of alcohol present; this will be $n/1000$, or $0.1n$ per cent.

"The recognition of the yellowish green tint is made much easier by having tubes with definite quantities of alcohol, such as 1/500th, 1/666th, 1/1000th, 1/1500th, 1/2000th, 1/3000th, for comparison. 50 c.c. is a convenient quantity to work with."

It is well known that in distilling mixtures of alcohol and water, the alcohol being present in such quantity as to be easily measured, the whole of the alcohol is supposed to come over in the first third of the distillate. We thought it would be interesting to see if very dilute solutions behaved in the same manner.

The following is the method we used:—

600 c.c. of the dilute solution (1/500th to 1/3000th) is placed in a 1-litre flask and distilled, 1/20th of the distillate, or 30 c.c., is collected; the operation is repeated with another 600 c.c., and 2/20ths of the distillate is collected. This is repeated a third time, and 3/20ths is collected, and so on. *A priori*, it would appear to be simpler to collect consecutive 1/20ths and to estimate the alcohol in each; but alcohol comes over so rapidly that the estimation of fractions over 2/20ths becomes difficult, and the errors of observation are increased.

The method we adopted is longer, but, on the other hand, is much more exact. Our experiments were carried out on mixtures of 1/500th, 1/1000th, and 1/3000th. The following table shows the results we obtained:—

Fractions of distillate collected.	Amount per cent of alcohol in the distillate.		
	1/500th.	1/1000th.	1/3000th.
1/20th	50	52	55
2/20ths	75	78	79
3/20ths	88	90	91
4/20ths	90	92	94
5/20ths	91	93	97
6/20ths	92	94	100
7/20ths	93	98	100
10/20ths	100	100	100

An examination of these figures shows that:—

1. The distillation of alcohol is relatively quick. The first 1/20th of distillate containing 50 per cent of the total quantity present.
2. The quantity per cent of total alcohol for a given fraction is greater as the mixture is more dilute.
3. In mixtures of 1/500th and 1/1000th the third part (fractions of 6/20ths and 7/20ths) does not contain all the alcohol present.
4. But with the mixture of 1/3000th we are not wrong in assuming that the alcohol is entirely contained in the first quarter of the distillate.

The direct consequence of these results was the attempt to estimate alcohol in solutions containing not more than 1/3000th to 1/10,000th. Two experiments made with solutions of 1/5000th and 1/10,000th gave, in the first quarter, distillates containing 1/1250th and 1/2500th, which, estimated by the method described above, gave us the total alcohol present.

It may be remarked that the size of the flask, in proportion to the amount of liquid distilled, did not appear under the conditions described to have any particular influence.—*Bull. de la Société Chimique de Paris*, xii.-xiii., No. 7.

Disinfection with Formic Aldehyd.—Referring to our note on this subject (*CHEM. NEWS*, lxxv., p. 251), we are informed that the Medico-Hygienic Inventions Co., Lim., are the sole agents for the apparatus known as Trillat's autoclave, used by Dr. Winter Blyth in his experiments on disinfection with formic aldehyd.

RELATIONS BETWEEN THE MELTING-POINTS AND THE LATENT HEATS OF FUSION OF THE METALS.*

By JOSEPH W. RICHARDS, Ph.D.

In a lecture before the Franklin Institute in January, 1893, on "The Specific Heats of the Metals," I announced the fact that in the case of most of the metals whose latent heats of fusion were known, this quantity bears a simple relation to the heat required to raise the metal from absolute zero -273° (C.) to its melting-point. In most cases the former is one-third the latter. I even ventured to predict that the latent heat of fusion of gold was about 14 calories, and it has since been determined by Roberts-Austen as 16.3. Further, several latent heats have since been determined which conform to the above relation, and I have thought it opportune to collect these data and point out the limits of the relation, with some other observations which later thought on the subject has developed.

In the following table there is given, first, the heat required to raise 1 kilogram of the metal from the absolute zero to its melting-point (using the most probable values for the specific heats and extrapolating to -273° , for a discussion of which data reference is made to the paper already quoted); second, a simple fraction of this quantity; and, lastly, the actually determined latent heats of fusion, the experimental errors of which are probably 5 to 10 per cent:—

Element.	Heat absorbed from -273° C. to the melting-point.		Latent heat of fusion. (Experimental)..
Sodium	107.8	$\frac{1}{3} = 35.9$	34.7
Aluminium	215.0	$\frac{1}{3} = 107.5$	100.0
Potassium	54.9	$\frac{1}{3} = 18.3$	15.7
Copper	145.3	$\frac{1}{3} = 48.4$	43.0
Zinc	71.2	$\frac{1}{3} = 23.4$	22.6
Gallium	21.9	$\frac{1}{3} = 7.3$	19.2
Palladium	125.0	$\frac{1}{3} = 41.7$	36.3
Silver	74.7	$\frac{1}{3} = 24.9$	24.7
Cadmium	30.7	$\frac{1}{3} = 10.2$	13.1
Tin	27.6	$\frac{1}{3} = 9.2$	14.5
Platinum	83.4	$\frac{1}{3} = 27.8$	27.2
Gold	45.3	$\frac{1}{3} = 15.1$	16.3
Mercury	7.5	$\frac{1}{3} = 2.5$	2.8
Lead	17.7	$\frac{1}{3} = 5.9$	5.4
Bismuth	14.4	$\frac{1}{3} = 4.8$	12.4

Of the fifteen cases, the relation in eleven cases is one-third (in almost every case within the limits of the experimental errors); in two cases the fraction is apparently one-half, and in two cases unity. That for ten cases the ratio should be so uniform, with latent heats ranging from less than 3 to nearly 50, is an indication of some intimate connection between these physical constants of the elements.

Regarding the exceptional cases it occurred to me that aluminium, tin, and bismuth are known to act anomalously in many relations, as if their molecular structures were different from that of the other metals. (We have no data from which to discuss gallium in these relations). For instance, in lowering the freezing-point of other metals, aluminium is known to act as if its molecular formula were double that of other metals in the molten state. In Pictet's observation of the connection between the melting-point, coefficient of expansion, and atomic volume of an element, bismuth and tin are among the chief exceptions.

* A Paper read before the Chemical Section of the Franklin Institute. From the *Journal of the Franklin Institute*, May, 1897.

[Pictet's rule is that the melting-points of the elements (T in absolute degrees) are, in many cases, inversely proportional to their coefficient of expansion by heat (α = linear expansion 0° to 100°C.) and to the relative distance

of their atoms apart ($\sqrt[3]{V}$, where V is the atomic weight divided by the specific gravity, or atomic volume) for which Pictet's relation is expressed by—

$$T \cdot \alpha \cdot \sqrt[3]{V} = 4.5$$

or—

$$T = \frac{4.5}{\alpha \cdot \sqrt[3]{V}}$$

In fact, the products of these three quantities are not exactly equal, but vary between 4 and 5, the reason being, doubtless, that the average specific gravity and rate of expansion from -273° to the melting-point varies somewhat from the gravity at 20° and rate of expansion at 0° to 100° as used in his calculations].

As already mentioned, bismuth and tin were Pictet's chief exceptions, and since they were anomalous in regard to their latent heat relations, I was led to compare these several relations among themselves, and to the following chain of reasoning:—Since the atomic heats of the elements (specific heat into atomic weight) at 20° to 100° are, by Dulong and Petit's law, approximately equal to 6.4, then, assuming that the average specific heat from -273° to the melting-point does not vary much from the figure for 20° to 100° , the heat in atomic weight of a metal at its melting-point is approximately 6.4 T; and, assuming the relation between the latent heat of fusion and the total heat in the metal at its melting-point as $\frac{1}{2}$, the latent heat of fusion of an atomic weight of a metal becomes approximately 2.1 T.

But we can at once connect this expression with Pictet's rule, and write:—

$$L = 2.1 T = \frac{4.5 \times 2.1}{\alpha \cdot \sqrt[3]{V}} = \frac{9.5}{\alpha \cdot \sqrt[3]{V}}$$

where L is the latent heat of fusion of an atomic weight of the metal.

To test the validity of this expression (which, for reasons already explained, cannot claim exact accuracy), we will take Pictet's values for $\alpha \cdot \sqrt[3]{V}$ (which are based on the best available data) and make the calculation for the metals whose latent heat of fusion and coefficient of expansion are both known.

	$L = \frac{9.5}{\alpha \cdot \sqrt[3]{V}}$	$\frac{L}{\text{At. wt.}}$	Latent heat experimentally obtained.
Aluminium ..	1900	70.4	100.0
Copper ..	3006	46.2	43.0
Zinc ..	1561	24.6	22.6
Palladium ..	3832	36.1	36.3
Silver ..	2541	23.5	24.7
Cadmium ..	1253	11.1	13.1
Tin ..	1712	13.7	14.5
Platinum ..	5106	26.3	27.2
Gold ..	3035	15.5	16.3
Mercury ..	654	3.3	2.8
Lead ..	1284	6.2	5.4
Bismuth ..	2777	13.4	12.4

Excepting aluminium, the coincidences are so close in the case of all the others that the calculated values in every case fall within the permissible limits of experimental errors, and it must be remembered that the above table contains all the metals for which the data are at present available. The non-metal sulphur expands so irregularly that no calculation can be made for it.

The closeness of the above coincidences may lead us to apply the formula to those other elements whose

coefficients of expansion are known, but whose latent heat has not yet been determined, and thus to predict approximately the probably value of their latent heat of fusion.

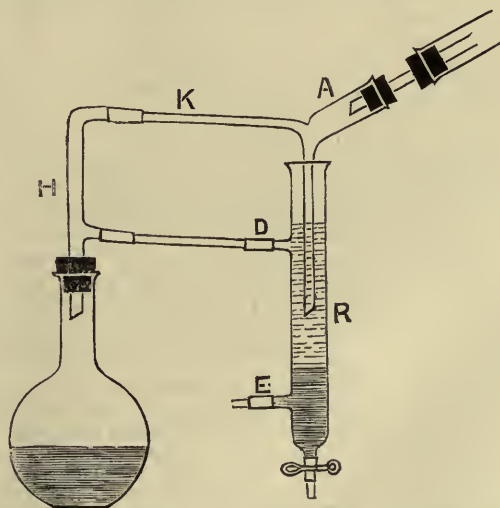
	Calories.
Magnesium ..	58
Pure iron ..	69
Cobalt ..	68
Nickel ..	68
Selenium ..	13
Ruthenium ..	46
Rhodium ..	52
Indium ..	8
Antimony ..	16
Tellurium ..	17
Osmium ..	35
Iridium ..	28
Thallium ..	5.8

In the case of those other elements whose coefficients of expansion and specific heat are both unknown, the latent heats of fusion may be predicted, approximately, simply from the melting-point, by using the relation $L = 2.1 T$. The above comparisons, however, have shown that the dependence of the atomic latent heat of fusion on the absolute temperature of the melting-point, or on the total heat in the metal at its melting-point, is less exact than the dependence on the coefficient of expansion and atomic volume, and we should give the latter relationship preference in predicting unknown latent heats.

APPARATUS FOR STEAM DISTILLATION.

By WM. CORMACK.

In using the apparatus for steam distillation described by Matthews in the *Journal of the Chemical Society*, vol. lxi., p. 318, it occurred to me that by a slight modification in the structure and arrangement of the parts a gain both in point of simplicity and freedom of construction might be secured. The modified apparatus, a sketch of which



is annexed, consists of a head-piece, H, an adapter, A, and a receiver, R. The head-piece, which resembles that of a Drechsel extraction apparatus, is fitted into the neck of the flask by means of a cork or of rubber tubing. The vapours from the flask pass along the tube K to the condenser through the adapter, A, which is an ordinary bent adapter with a short tube sealed on at the bend. After

condensation, the liquid runs down the vertical tube of the adapter into the receiver, R. As shown in the figure, the apparatus is set up so as to collect a liquid which is heavier than water. In this case the liquid sinks to the bottom of the receiver, and the water floats on the top. As soon as the level of the water has attained a certain height it flows back through the upper side-tube, D, into the flask. It is essential that D should be at a lower level than the lower side-tube of H. The receiver is drawn out at the bottom and furnished with a rubber tube, clip, and jet, so that it may act at the same time as a separator. The lower side-tube, E, of the receiver is closed by a cap of rubber and glass rod.

If the liquid to be collected is lighter than water, the upper tube, D, is closed by the cap, and connection made by glass and rubber tubing between the lower tube, E, and the head-piece. The water then sinks to the bottom, and flows back into the flask through the lower tube and its connections.

The chief points in which this modification differs from the original apparatus of Matthews, is that there is no fast connection between the condenser and the receiver, but merely a water-joint, and that the same apparatus may be used for collecting liquids which are heavier or lighter than water. I have found the apparatus very satisfactory, so far as I have tested it, especially when small quantities of substances are being dealt with. The liquids in the receiver become warm after a time, which may or may not be advantageous, according to the nature of the substance. If cooling is required, this is best done by jacketing the receiver.

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EXPLANATION OF SOME EXPERIMENTS OF G. LE BON'S.

By H. BECQUEREL.

THE *Comptes Rendus* of one of our last sessions contains a paper (April 20th, 1897), by M. Perigot, in which the author, in giving account of certain experiments by G. Le Bon, after some very judicious remarks, invokes the transparency of ebonite for white light. The fact of the transparency of the ebonite plates used in these experiments for the active radiations is undeniable, but I purpose showing that the phenomena observed are not due to what we call *white light*,—that is to say, to the radiations employed most generally in photography,—but to the red radiations from the least refrangible extremity of the spectrum, and the ultra-red radiations for which ebonite is very transparent.

An experiment which M. G. Le Bon has described, in reply to M. Perigot, leaves me no doubt in this respect, and since Monday last I have indicated the following explanation to our colleagues MM. d'Arsonval, Lippmann, and Poincaré. I have verified its correctness the next day by several experiments.

This is, in the first place, what constitutes the experiments of M. Le Bon to which I have just alluded. We take a surface covered with phosphorescent zinc sulphide, and expose it to the light; then we cover it with a plate of ebonite, upon which we arrange as screens various objects,—e. g., coin,—and expose the whole to the sun for some seconds. On examining afterwards, in the dark, the surface of the zinc sulphide (originally phosphorescent), we find that it is almost extinguished, except under the piece of metal where the phosphorescence is still very visible. M. Le Bon thought himself entitled to conclude from this experiment that the metal sent out rays which excite phosphorescence. The explanation is quite different. These red and ultra-red rays sent by the sun traverse the ebonite, and, as it has been known long ago, extinguish the phosphorescence over the whole luminous surface,

except at the points protected by the metallic screen; at these points the phosphorescence obtained by the previous illumination fades very slowly. I have not proposed to determine what are the radiations transmitted through the ebonite. I will recall that it results, from the very ancient experiments of my father and from those which I have had occasion to publish, that the red and ultra-red rays determine a rapid extinction and the phosphorescence of bodies previously illuminated, an extinction generally preceded by a temporary excitement which is not visible with phosphorescent zinc sulphide or hexagonal blende. If we project a spectrum upon the surface of a phosphorescent sulphide, the ultra-red region appears in black upon a luminous ground, in consequence of the extinction set up by the corresponding radiations; and I have shown that this extinction presents maxima and minima, variable not only with the nature of the luminous source, but with the nature of the phosphorescent substance.

For hexagonal blende the spectrum of extinction extends from the red of the visible region to the wave-length $1\mu 5$, presenting a minimum about $1\mu 1$ and $1\mu 2$, and a strong maximum between $1\mu 3$ and $1\mu 4$.

If we project upon a screen of hexagonal blende, previously illuminated, a spectrum obtained with the solar radiations having traversed a plate of ebonite 0.6 m.m. in thickness, we note the immediate appearance of the infra-red extinction-band of 1.3μ to 1.8μ ; then, after a few instants, we see appear, rather less intense, the extinction between the wave-lengths $1.3\mu 2$ and the extreme visible red near A.

On concentrating the solar light with a lens we perceive red rays near the extreme visible red of the solar spectrum.

Ebonite is therefore transparent for the radiations which extinguish phosphorescence of zinc sulphide, and the explanation of Le Bon's experiment is evidently that which I have given above.

If in this experiment we form the screen of a substance which arrests the ultra-red rays, as the red rays transmitted are much enfeebled, the silhouette of the screen appears luminous on an extinguished ground. If the screen is formed of a diathermous substance like rock-salt, the totality of the phosphorescent surface is extinct. A red glass, or any substance transmitting the ultra-red rays and arresting the luminous rays,—blue, violet, and ultra-violet,—which excite the phosphorescence of blende, will give the same results if substituted for ebonite.

This transparency, perfectly demonstrated, enables us to explain in all their details the photographic experiments of M. Le Bon through ebonite.

It is known that my father showed, as early as 1840, that a photographic plate insensible to the yellow and the red rays, becomes sensitive to these rays and even to the ultra-red rays if it is exposed for a short time to light, i. e., slightly veiled. The red and ultra-red rays continue the action commenced by the white, blue, or ultra-violet light.

This phenomenon, discovered with Daguerre plates, is manifested with most sensitive photographic surfaces, and in particular with plates of silver gelatino-bromide.

In the experiments of M. Le Bon a photographic plate, previously veiled (the condition essential to the success of the experiments), is exposed under a plate of ebonite to the action of the solar rays. Metallic screens placed on the ebonite mark their silhouette when we come to develop the plate. Under these conditions the photographic plate undergoes through the ebonite the continuing action of the red and ultra-red rays. The metallic screens protect the plate against this action; if the exposure has been relatively short, the ground appears on development darker than the silhouette of the screens; if the exposure has been prolonged, it results that, in consequence of a phenomenon of reversal well known, the background appears less affected than in the region protected by the screen.

The active rays in these experiments of continuation

are the extreme red rays near A. As these radiations are much weakened by the ebonite, the exposure must be relatively very long, and the phenomena are not as distinct as with the highly sensitive gelatino-bromide.

The same phenomena are obtained on substituting red glass for ebonite.

I will add that a plate of ebonite of 0.6 m.m. in thickness, if examined with Melloni's apparatus, transmits 0.04 per cent of the dark heat emitted by a plate of copper at 400°, and arrests almost entirely the radiation from a source of heat at 100°.

In sum, the phenomena which M. G. Le Bon endeavours to ascribe to an assumed *black light* of an unknown nature are simply effects produced by the red or ultra-red rays, the principal properties of which have been well known for more than fifty years.—*Comptes Rendus*, vol. cxxiv., p. 984.

RESEARCHES ON THE BIOLOGICAL ACTION OF THE X-RAYS.

By J. SABRAZES and P. RIVIERE.

The apparatus used in our experiments consisted of a bi-anodic focus tube (M. Segny's design), excited by powerful Ruhmkorff coil, yielding a current of 6 ampères and 16 volts, sparks of 35 c.m. in length. With the aid of this tube we obtained radiographs at the distance of 3.50 metres and behind a screen of boards. This arrangement evidently permitted the easy exploration of the limbs and of the thorax.

The objects studied were arranged at about 15 c.m. from the fluorescent source; they were enclosed in black paper to protect them from luminous radiations, and were placed in metallic communication with the earth.

1. Experiments on *Microbacillus prodigiosus*.

We proposed to study the action of the X-rays on a microbe peculiarly sensitive to changes brought on in the physico-chemic conditions of its development, and apt to translate this sensitiveness by modifications permanent and easy of observation.

The *Microbacillus prodigiosus* answers to these requirements, especially if we address ourselves, as we have done, to a race eminently chromogenous.

If we cultivate this microbe at a dysgenic temperature of 37°,—if we modify the reaction of the medium by alkalisating it strongly,—if we diminish the access of atmospheric oxygen,—if we expose the culture to the solar radiations, to the action of an injurious substance, e.g., of an antiseptic,—the pigmentary properties are attenuated and effaced progressively, and in time, although the colonies continue to grow, they remain more or less colourless. We know also that it is sufficient to acidify the culture-bouillon, or to raise the temperature of the stove to 50°, to modify profoundly the aspect of the microbial cells which become filamentary or even spiriform. It will therefore be easy to appreciate the modifications impressed on this microbe by bringing into play a novel biological condition; it will be sufficient to examine the oscillations of the chromogenic power and the morphologic variations of the cellulæ as compared with type cultures in a long series of successive generations.

We have proceeded as follows:—The cultures, of a fine carmine-red, were collected on gelose, and arranged in the form of a small pointed heap, in the middle of a sterilised watch-glass wrapped in black paper. The X-rays act directly through the paper upon the seed, the cavity of the watch-glass being placed opposite the phial.

We operate exactly in the same manner with a check-culture, except that the watch-glass covered with black paper was screened from the X-rays.

Daily for twenty days we caused the X-rays to act upon the microbe for an hour each day.

In spite of the power of our phial, the number of passages, and the duration of exposure to the X-rays, we have not observed any appreciable modification of the *Microbacillus prodigiosus*, either in its chromogenic properties or in its morphological characters or its vegetability. This microbe has shown itself indifferent to Röntgen's rays.

2. Experiments on *Leucocytes*.

Two frogs, as nearly as possible of the same bulk, were fixed upon cork in the ordinary manner.

After cauterisation with the thermo-cautery, so as to avoid any loss of blood, we introduced—at a point of the leguments chosen at the level of the mean part of the abdomen—at the aperture thus made, a pointed tube, previously washed with a culture of microbes yielding products of positive chemiotaxis.

We exposed one of the frogs to the X-rays after having taken the precautions mentioned above. The other, which served as a check-type, was withdrawn from the influence of the radiation by a metallic enclosure. We prolonged the experiment for several hours, and at the end of the time collected the liquid which had transuded into the tubes. We counted the *leucocytes* existing in the lymph thus collected, and examined with the microscope the figured elements held in suspension.

From the numerous experiments thus made the conclusion follows that the X-rays do not interfere with the efflux of the white globules; their number is sensibly the same in both cases. Phagocytosis is equally effected. In some experiments even the quantity of lymph emanating from frogs submitted to the X-rays was slightly greater than that in the check-tubes.

3. Action of the X-rays upon the Heart.

Some observers have detected in man cardiac disturbances following on application of the X-rays.

The tracings of the heart of a frog placed below a powerful source of X-rays showed us that the rhythm of this organ was not modified in its periods even after an exposure of more than one hour.—*Comptes Rendus*, vol. cxxiv., p. 979.

DETERMINATION OF PHOSPHORUS IN CHEMICO-LEGAL INVESTIGATIONS.

By M. SPICA.

THE author proceeds as follows:—The substance in question is divided into three parts, of which one is reserved for check-experiments; a second serves for the qualitative detection of phosphorus by Mitscherlich's process; and the third for the quantitative determination of phosphorus. For the last purpose he employs the free phosphorus, or that present in the direct volatile state, according to the procedure of R. Fresenius and Newbauer, which he has somewhat modified. The flask containing the material is closed with a stopper having three perforations for the usual entrance and exit of gas, and for a tube-funnel closed with a glass cock. To the flask is connected a tubulated receiver, which is again connected with three Peligot tubes containing neutral solutions of silver nitrate. After the transmission for six to eight hours of carbonic acid washed in solution of silver nitrate, it is tested anew by fresh tubes filled with silver solution, to determine if phosphoriferous substances pass over on a further passage of carbonic acid.

If this is not the case we proceed to determine those compounds of phosphorus which are not reduced by nascent hydrogen, for which purpose the author uses the procedure of Dusart as modified by Blondlot. After the contents of the flask have been allowed to cool in the current of carbonic acid, the purest zinc is rapidly introduced into the flask, which is then closed, and sulphuric acid is run in through the funnel tube until there sets in

a slight development of hydrogen, which is kept in very slow action for five to six days. After this lapse of time we satisfy ourselves as above of the completion of the reaction.

For the ultimate gravimetric determination of the phosphorus, the author oxidises the liquid found in the receivers with nitric acid, and determines the phosphorus in the solution as ammonium phospho-molybdate.—*Zeit. Anal. Chemie*, xxxvi., p. 347.

COLORIMETRIC DETERMINATION OF SMALL QUANTITIES OF NITROUS ACID.

By Prof. Dr. E. RIEGLER.

IN connection with my communication on the detection of nitrous acid with naphthionic acid, I point out that by means of this reaction small quantities of nitrous acid can be easily determined colorimetrically. We dissolve 0.406 grm. pure dry silver nitrite in hot water, add sodium chloride in slight excess, and dilute after cooling to 1 litre. After the deposit has subsided we take 100 c.c. of the clear solution, and dilute again to 1 litre with distilled water. Of this last solution we put 100 c.c., corresponding to 0.001 grm. N_2O_3 , into a flask, add a small penknife-point (about 0.05 grm.) full of crystalline naphthionic acid and 5 or 6 drops of concentrated hydrochloric acid; shake up well, and add 30 drops of concentrated ammonia (liquid). After thorough shaking we have a rose-coloured liquid, the tint of which serves as a standard for the above concentration. In order to find the proportion of nitrous acid in a water, we place 100 c.c. of the water in a flask, add a small penknife-point full of naphthionic acid, shake well up, and then add 30 drops of concentrated liquor ammoniac. We then compare, by means of the colorimeter, the intensity of this solution with that of the standard, and calculate according to known rules the quantity of the nitrous acid.

In this manner 0.00001 grm. N_2O_3 can be determined in 100 c.c. of water.

In case the water in question contains more than 0.001 grm. N_2O_3 in 100 c.c., it must be suitably diluted with distilled water before determination.—*Zeit. Anal. Chemie*, xxxvi., p. 306.

FOURTH ANNUAL REPORT OF COMMITTEE ON ATOMIC WEIGHTS.

RESULTS PUBLISHED IN 1896.*

By F. W. CLARKE.

DURING 1896 the activity in the determination of atomic weights was only moderate. Comparatively few papers on the subject appeared, but some of these were of excellent quality. The question is often asked, Why are new determinations important? Are not those we have good enough for all practical purposes. To this question I have an interesting answer, such as has not hitherto been published.

There are two rival values for the atomic weight of chromium. One, 52.5 approximately, based on the old work of Berlin, is still used by European analysts. The other, 52.1, depends upon later and more accurate researches, and is used in the United States. Mr. William Glenn, of the Baltimore Chrome Works, informs me that that establishment imports chrome iron ore by the shipload, the value being determined by a volumetric assay in which the atomic weight of chromium is involved. It is assayed in Glasgow with the older value for chromium,

	H = 1.	O = 16.
Aluminum..	26.91	27.11
Antimony ..	119.52	120.43
Argon..	?	?
Arsenic ..	74.44	75.01
Barium ..	136.39	137.43
Bismuth ..	206.54	208.11
Boron ..	10.86	10.95
Bromine ..	79.34	79.95
Cadmium ..	111.10	111.95
Cæsium ..	131.89	132.89
Calcium ..	39.76	40.07
Carbon ..	11.92	12.01
Cerium ..	139.10	140.20
Chlorine ..	35.18	35.45
Chromium..	51.74	52.14
Cobalt ..	58.49	58.93
Columbium ..	93.02	93.73
Copper ..	63.12	63.60
Erbium ..	165.06	166.32
Fluorine ..	18.91	19.06
Gadolinium ..	155.57	156.76
Gallium ..	69.38	69.91
Germanium ..	71.93	72.48
Glucium ..	9.01	9.08
Gold ..	195.74	197.23
Helium ..	?	?
Hydrogen ..	1.000	1.008
Indium ..	112.99	113.85
Iodine ..	125.89	126.85
Iridium ..	191.66	193.12
Iron ..	55.60	56.02
Lanthanum ..	137.59	138.64
Lead ..	205.36	206.92
Lithium ..	6.97	7.03
Magnesium ..	24.10	24.28
Manganese ..	54.57	54.99
Mercury ..	198.49	200.00
Molybdenum ..	95.26	95.99
Neodymium ..	139.70	140.80
Nickel ..	58.24	58.69
Nitrogen ..	13.93	14.04
Osmium ..	189.55	190.99
Oxygen ..	15.88	16.00
Palladium ..	105.56	106.36
Phosphorus ..	30.79	31.02
Platinum ..	193.41	194.89
Potassium..	38.82	39.11
Praseodymium..	142.50	143.60
Rhodium ..	102.23	103.01
Rubidium ..	84.78	85.43
Ruthenium ..	100.91	101.68
Samarium ..	149.13	150.26
Scandium ..	43.78	44.12
Selenium ..	78.42	79.02
Silicon ..	28.18	28.40
Silver..	107.11	107.92
Sodium ..	22.88	23.05
Strontium ..	86.95	87.61
Sulphur ..	31.83	32.07
Tantalum ..	181.45	182.84
Tellurium ..	126.52	127.49
Terbium ..	158.80	160.00
Thallium ..	202.61	204.15
Thorium ..	230.87	232.63
Thulium ..	169.40	170.70
Tin ..	118.15	119.05
Titanium ..	47.79	48.15
Tungsten ..	183.43	184.83
Uranium ..	237.77	239.59
Vanadium ..	50.99	51.38
Ytterbium ..	171.88	173.19
Yttrium ..	88.35	89.02
Zinc ..	64.91	65.41
Zirconium..	89.72	90.40

* *Journal of the American Chemical Society*, xix., No. 5.

and in Baltimore with the modern datum. A cargo amounts to about 3500 tons; and the difference in price due to the difference between 52.1 and 52.5 for chromium amounts to about 367.50 dols. per shipload. This difference is large enough to show the importance of accurately determined constants from a commercial point of view, and suggests that other similar cases might be found by a careful scrutiny of our analytical processes.

My own "Recalculation of the Atomic Weights," a new edition of the work published originally in 1882, is now complete and in the printer's hands. It will probably be published early in 1897, and the appended table of values represents the results obtained by combining all the best data.

The following new determinations of atomic weights represent the work published during the year:—

Oxygen.—In the report for 1895, J. Thomsen's gravimetric measurements of the H:O ratio were cited. Early in 1896 the same chemist (*Zeit. Anorg. Chem.*, xii., 4), by a novel method, determined the ratio of densities. First, he found the volume of hydrogen in litres, liberated by the solution of one grm. of aluminum, to be as follows:—

1.24297
1.24303
1.24286
1.24271
1.24283
1.24260
1.24314
1.24294

Mean .. 1.24289 $\pm$ 0.00004

In his earlier research Thomson found the weight of hydrogen corresponding to 1 grm. of aluminum to be 0.1190 $\pm$ 0.000015 grm. Hence 1 litre of hydrogen at 0°, 760 m.m., and 10.6 metres above sea-level, is 0.090032 $\pm$ 0.000012 grm.; or at sea-level in latitude 45°, 0.089947.

For the volume of one grm. of oxygen at 0°, 760 m.m., and at Copenhagen, Thomsen found, in litres:—

0.69902
0.69923
0.69912
0.69917
0.69903
0.69900
0.69901
0.69921
0.69901
0.69922

Mean .. 0.69910 $\pm$ 0.00002

At sea-level, latitude 45°, 0.69976 $\pm$ 0.00002.

Hence one litre weighs 1.42906 $\pm$ 0.00004 grm. Dividing this by the value found for hydrogen we have for the ratio desired—

15.8878 $\pm$ 0.0022.

Correcting this by the known data for the volumetric composition of water we get—

O = 15.8690 $\pm$ 0.0022,

a value identical with that found gravimetrically, and very close to the measurement by Morley.

Silver.—The atomic weight of silver has been determined electrolytically by Hardin (*Fourn. Am. Chem. Soc.*, xviii., 990; *CHEM. NEWS*, lxxv., 28 *et seq.*). The nitrate, acetate, and benzoate, mixed in aqueous solution with solutions of pure potassium cyanide, were electrolysed in platinum dishes. The data are as follows, with vacuum weights, and reduced with N = 14.04, C = 12.01, H = 1.008, and O = 16.

Nitrate Series.

Weight AgNO ₃ .	Weight Ag.	Atomic wt. Ag.
0.31202	0.19812	107.914
0.47832	0.30370	107.900
0.56742	0.36030	107.923
0.57728	0.36655	107.914
0.69409	0.44075	107.935
0.86367	0.54843	107.932
0.86811	0.55130	107.960
0.93716	0.59508	107.924
1.06170	0.67412	107.907
1.19849	0.76104	107.932

Mean .. 107.924

Acetate Series.

Weight salt.	Weight Ag.	Atomic wt. Ag.
0.32470	0.20987	107.904
0.40566	0.26223	107.949
0.52736	0.34086	107.913
0.60300	0.38976	107.921
0.67235	0.43455	107.896
0.72452	0.46830	107.916
0.78232	0.50563	107.898
0.79804	0.51590	107.963
0.92101	0.59532	107.925
1.02495	0.66250	107.923

Mean .. 107.922

Benzoate Series.

Weight salt.	Weight Ag.	Atomic wt. Ag.
0.40858	0.19255	107.947
0.46674	0.21999	107.976
0.48419	0.22815	107.918
0.62432	0.29418	107.918
0.66496	0.31340	107.964
0.75853	0.35745	107.935
0.76918	0.36247	107.936
0.81254	0.38286	107.914
0.95673	0.45079	107.908
1.00840	0.47526	107.962

Mean .. 107.938

The mean of all three series is—

Ag = 107.928.

This value agrees well with the values found by Stas and by Marignac, and so creates a presumption in favour of the electrolytic method, which Hardin has also applied to determining the atomic weights of mercury and cadmium.

(To be continued).

SOME PRESENT POSSIBILITIES IN THE ANALYSIS OF IRON AND STEEL.*

By C. B. DUDLEY.

(Concluded from p. 270).

SOME years ago, with the publication (*Trans. Am. Inst. Mining Eng.*, ix., 397) of what is commonly known as Ford's method, the determination of manganese took a decided step forward, at least in this country, so far as speed is concerned. Previous to that time the long and laborious acetate method, which involved the separation of the iron from the manganese as basic acetate and subsequent precipitation of the manganese by means of bromine or as pyrophosphate, had held full sway.

* Presidential Address delivered at the Troy Meeting of the American Chemical Society, December 29, 1896. From the *Journal of the American Chemical Society*, xix., No. 2.

Ford's contribution consisted, as is well known, in separating the manganese from hot nitric acid solution of the iron or steel, by means of potassium chlorate, and Williams (*Trans. Am. Inst. Mining Eng.*, x., 100) added the modification, now in common use, of determining the separated oxide of manganese, by its action on a standard solution of ferrous sulphate or oxalic acid. This method, as now worked in many laboratories, gives a single result in forty minutes and two in an hour, and enables one operator to turn out twenty to twenty-five determinations in a day. The accuracy of this method has been questioned. We are not aware of any recent symposium on manganese, where different chemists using different methods have worked on the same steels. In our hands this method gives results closely agreeing with check work done by the more laborious and generally accepted accurate methods, provided the sample contains not more than three-fourths of a per cent. On samples containing over 1 per cent of manganese the results are apt to be low, owing probably to the fact that the manganese does not separate from the nitric acid solution as manganese dioxide, but as some other oxide whose composition is not positively known. In the calculation it is customary to regard the separated oxide as manganese dioxide, and this leads to perceptible error on large amounts. Producers and consumers rarely contend much over manganese in steel, and methods for its determination have perhaps not received, on that account, all the attention they deserve. There is evident need of more work on this subject.

The methods for the determination of silicon can hardly be regarded as in a perfectly satisfactory condition. If evaporation to dryness to render silica insoluble is employed, the time required is considerable. If dehydration by means of sulphuric acid and heat, as suggested by Drown (*Trans. Am. Inst. Mining Eng.*, vii., 346) is employed, there are difficulties which interfere somewhat with accuracy. There seems little doubt but that in skilled hands, with sufficient care taken in the manipulation, a couple of determinations may be made on the same sample, using Drown's method, that will agree closely with each other and with results given by the longer and more laborious methods. On the other hand, where one operator is making a number of determinations at the same time there is much danger of error, due either to failure to dehydrate sufficiently or to over-heating, resulting in the formation of insoluble iron salts. Our experience indicates that the margin between these two extremes is not very wide, and that it is fully as frequent to have duplicates on the same sample disagree as to agree. Our observations point to the view that the difficulty of insufficient dehydration is due to the separation of iron salts as the sulphuric acid concentrates. These salts enclose gelatinous silica, and prevent the dehydrating acid from getting at it. Unless great pains are taken, therefore, to secure this contact by sufficient stirring, the results will be low. If by some modification the iron salts could be kept in solution until the silica is rendered quite insoluble, it would apparently be a decided step forward with this method. It may not be amiss here to call attention to the fact first noticed in the laboratory of the Pennsylvania Railroad Company,* that after the dehydration and subsequent dilution are finished, if an interval of a few hours is allowed to elapse before filtration, the silica will re-dissolve and the results be low. Apparently, as we are able to work the method, the silica is not completely dehydrated, but only sufficiently so that if filtered at once fairly accurate results will be obtained.

It is difficult to say anything positive about the speed and output of Drown's method. It is probably safe to say that a couple of determinations could be made in an hour and a half, but, on account of the difficulty men-

tioned above, the method does not lend itself well to working on a large number of samples at once, and consequently a large daily output is somewhat interfered with.

It must also be said of the methods for the determination of sulphur in iron and steel, that those most in use are hardly as satisfactory as could be desired. The studies of Phillips (*Journal of the American Chemical Society*, xvii., 891) conclusively show that when using the evolution method the whole of the sulphur content is not given off in such a form as to be retained by the usual means employed to catch the gas. It seems not too much to say that it is hazardous to use the evolution method on pig or cast iron, even when fusion of the residue is employed. The formation of unoxidisable gases containing sulphur, in the application of the evolution method to steel, has not, so far as our knowledge goes, yet been demonstrated, and accordingly the evolution method is still used largely on steels. But on pig and cast irons the oxidation method seems the only one applicable, and some recent studies of Blair, described in a paper at this meeting (see *Journ. Amer. Chem. Soc.*, xix., 114), indicate that on certain pig irons all the sulphur is not given, even by this method, unless the graphitic residue is fused with sodium carbonate and nitre. Both methods are somewhat slow, and there is need of further study. If some means could be found by which barium sulphate could be readily and accurately converted into sulphide, so that a volumetric method could be applied to this sulphide, it would be a decided step forward. The necessity in accurate work for purifying barium sulphate, as first obtained from almost any solution, by fusion and re-precipitation, adds quite considerably to the time required. With steels and two sets of evolution apparatus, using bromine for oxidation, two determinations may be made in two hours. With four sets of evolution apparatus, one operator can make twelve determinations in a day. In these cases purification by fusion is not attempted. By the oxidation method on pig or cast iron, two determinations require about five hours, while one operator with a supply of borings ahead and sufficient appliances, can get from ten to twelve results in a day. With this output purification by fusion is not attempted. If this is done, the time for a pair of determinations must be extended an hour and a half, and the daily output would be cut down at least a third.

From what has preceded in this hasty and necessarily imperfect survey of a portion only of the analytical methods in use in the iron and steel industry, it is clearly evident that there still remains an enormous amount of work to be done in connection with methods. We have touched upon only five of the fifteen or twenty constituents occurring in and affecting the quality of iron and steel, and find the methods for determining even those more or less imperfect, and needing more work. What will be our condition as chemists if, as seems probable, nickel, chromium, aluminum, tungsten, and the gases, oxygen, hydrogen, and nitrogen, either free or combined, within the next few years, come into prominence as constituents of iron and steel, and are made elements in important commercial contracts? Still further, thus far our methods are concerned almost entirely with the total content of the various constituents we are determining. We know very little about the compounds of the various constituents occurring in iron and steel, with the metal or with each other. Is the phosphorus present as phosphide or phosphate, or both? How besides as sulphide does the sulphur occur? Do the various carbides which are revealed by the microscope, and which are believed to be so closely dependent on the heat treatment which steel receives, and which are so intimately related to the value of the metal, differ from each other in carbon content, or only in crystalline form? Who will be the first to isolate any of these carbides? Who will first give us a practicable, accurate, and sufficiently rapid method for determining oxides in steel? Who will first completely investigate the relation between the chemistry and the chilling properties

* Address to the members of the Chemical Section of the Engineers' Society, at Pittsburgh, September 27th, 1892, by C. B. Dudley, on "Discrepancy in Chemical Work by Different Workers."

of cast iron? And who will first give us a study on the form in which nitrogen occurs in this metal, and a sufficiently rapid and accurate method for its determination? Truly the harvest of chemical work before us in connection with iron and steel is bounteous. Will the labourers be forthcoming to gather the harvest?

NOTICES OF BOOKS.

Water and Public Health. The Relative Purity of Waters from Different Sources. By JAMES H. FUERTES, Member of the American Society of Civil Engineers. First Edition. First thousand. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1897. Pp. x-75. 12mo., Ill.

IN this little volume the author has grouped the principal cities of the world into classes according to the quality of their public water supplies, and has made a comparative study of their mortality statistics. This statistical method of treatment makes the importance of pure water stand out in bold relief.

The author admits the uncertainty resulting from incorrect reports of health boards and physicians, as well as from unreliable figures of population; but he assumes that "in all large cities these causes of error are perhaps equal," and the inaccuracies will counterbalance so as not to seriously influence the general deductions that may be drawn.

The four chapters of the book are entitled:—I. Etiology and Prophylaxis of Typhoid Fever. II. When does Pure Water pay? III. Sanitary Value of Impounded and other Supplies. IV. Conclusion. Following this are four Appendices, containing much statistical information.

The work is illustrated by no less than seventy diagrams, showing in a graphic way the relations of pure water and foul water to public health in a large number of cities in Europe and America.

The author's conclusions are those of a common-sense view of the subject, based upon the facts presented.

It is surprising to learn that the important commercial city of Baltimore, having a population of 435,000, "has as yet no sewerage," and "the house-drainage is disposed of largely into cesspools and outhouses."

The author discusses the outbreaks of cholera and typhoid fever in Hamburg, Germany, and it is truly appalling to read the terrible figures of mortality in that unhappy city during the epidemic years.

The author believes that "properly designed and operated filters may be relied upon to purify any waters at present used for a public water-supply in the United States."

In Chapter II. the author treats of the question "When does pure water pay?" and discusses in a cold-blooded way the economic value of an individual to the community, and the amount of money which the community is authorised to expend to accomplish a reduction in the death-rate. He finds that the "community can afford to invest about three thousand dollars for every death forefended."

The Index to the volume is unusually full.

H.C.B.

Ninth Annual Report of the Agricultural Experiment Stations of the Louisiana State University for 1896. Baton Rouge. 1897.

AT the Audubon Park Station sugar-cane has been both the chief crop, and the chief study in the laboratories. In the field, experiments covering the entire subject of proper fertilisation of the cane have been continued. Physiological investigations of the sugar-cane, both in the field and under the microscope, have been energetic-

ally pushed during the year, and valuable results obtained.

The young citrus grove continues to grow and increase, and hardy growths of oranges, such as the Satsuma and Kumquat, grafted on the *trifoliata* stock, have resisted the most intense cold ever known in the neighbourhood.

The fibre plants, such as jute, hemp, and ramie, have also been successful, but unfortunately no trials of decorticating by machinery were made.

At No. 2 Station, Baton Rouge, as at the others, special attention was given to the growth of Egyptian cotton; the crops have been gathered in, and now await the arrival of a cotton gin from England. We should have thought it cheaper to send the cotton to England, as other people do.

Station No. 3, at Calhoun, has been unfortunate; the drought was so severe and prolonged that everything has been eaten up by the stock, and all the experiments vitiated.

Bulletin of the Agricultural Experiment Station. Second Series, No. 46. Leguminous Root Tubercles, by Prof. W. R. DODSON. Baton Rouge. 1897.

IT has long been known that leguminous plants are restorative in their character, when used for improvement of soils in a system of rotation of crops. Some years since it was discovered that the chief virtue of these plants was in fixing the nitrogen from the air, which was due to the tubercles which occur upon their roots. With the object of throwing more light on this subject, a number of experiments were made by Prof. Dodson.

When thin slices of these tubercles are examined with a high-power microscope, they are found to be filled with myriads of organisms resembling bacteria. It has been shown that when plants are cultivated in pots, under conditions where contact with these organisms is prevented, the accumulation of nitrogen in the plant is only equal to what is lost by the soil; but in the opposite case, when these organisms are present, the increase of nitrogen can only be accounted for by the assumption that the free nitrogen of the air has been used.

To find the influence of deep and shallow planting on the tubercle formation of the roots, experiments were made on cow peas, pea-nuts, garden beans, &c. Seeds were planted at a depth of 1, 2, 3, 4, 5, and 6 inches. After eight weeks the soil was thoroughly softened and carefully washed from the roots. Planting at 2 or 3 inches seemed to give the maximum roots that spread near the surface, and the greatest number of tubercles. The observations made in these plantings were supplemented by an examination of all the leguminous plants found in the vicinity, and it was shown that the nature of the soil had much to do with the depth at which the tubercles are well developed; they are deeper in sandy than in clayey soil, and deeper where deep cultivation has been practised than where shallow cultivation prevailed.

In the second set of experiments it was sought to obtain an approximate idea of the depth to which the nitrifying organisms penetrate, and find conditions favourable to their development. Pots containing sterilised soil were planted with several kinds of seeds,—clover, lima bean, lupins, &c.,—and watered with nutritive solution; but all grew very badly, and it was eventually found, by modifying the experiments somewhat, that though the organisms are found at a depth of 1 foot, they are not very abundant, and in no case was the infection so general by watering as from a surface inoculation.

Other experiments show that each plant, or at most, each genus of plants, will support but one kind of parasitic organism capable of developing the tubercles on its roots. For instance, in order that tubercles may be developed on alfalfa, a particular organism must be in the soil, and any quantity of cow peas, or other leguminous plants, will not furnish that organism. The cow pea likewise has its own peculiar parasite, and so on with the others. Yet dozens

may be grown side by side in the same soil, and each will develop its own tubercles.

Chemistry of Artificial Colouring-matters. ("Chimie des Matières Colorantes Artificielles"). By A. SEYEWETZ and P. SISLEY. (Part 5). Pp. 821. Paris: Masson and Co. 1897.

THE fifth and final instalment of this work has just appeared, and we must confess to a sense of great disappointment. The whole book has up to the present been produced in an excellent manner, beyond the style of the ordinary French book, both as regards printing and quality of paper used; but with this last number before us we cannot help thinking of Mark Twain's description of one of his own humorous sketches, which, he explains, is only a study in black and white, not a finished picture. The present work is decidedly a study; but when we come to twelve large pages of *errata*, comprising no fewer than 128 corrections, many of them apparently important ones, we feel more as if we were reading proof sheets than a published book.

We hope in a future edition the work will be sent out more fitted for the reviewer.

Papers and Notes on the Genesis and Matrix of the Diamond.

By the late HENRY CARVILL LEWIS, M.A., F.G.S. Edited from his unpublished MSS., by Professor T. G. BENNEY, D.Sc., LL.D., F.R.S. London, New York, and Bombay: Longmans, Green, and Co.

IT is indeed a mournful story told by the Editor in his Preface,—first the sudden death of the author, Prof. H. C. Lewis, in July, 1888, followed soon after by that of Prof. G. H. Williams, to whom the MSS. had been handed for publication. It is a matter of great satisfaction that it should have fallen to Prof. Bonney, who had the privilege, as he himself informs us, of hearing the papers read by the author at the meetings of the British Association, in 1886 and 1887, to prevent the valuable and interesting contributions to our knowledge of diamond-bearing rocks being lost to Science. The present time is very opportune for their publication, when the genesis and history of the diamond, brought into prominence by the researches of Prof. Moissan and others, is occupying considerable attention in the scientific world.

The book is small, well printed, and the illustrations—particularly those of a fragment of diamond-bearing rock from Kimberley and the micro-photographs of sections—are very good. The care shown by the late Prof. Lewis in the collection of his notes is very apparent, and it is not often that we are fortunate enough to find so much solid matter in so small compass.

The book is divided into three sections. The first—"On a Diamond-bearing Peridotite and on the History of the Diamond"—contains illustrations of sections of the diamond-bearing "pipes" at the de Beer's mine, and, in support of the suggestion that the gabbro or euphotide in which the diamonds are found is the "mother rock," the well-known fact is quoted that each of the several pipes furnish diamonds distinct in character, easily distinguishable from the others; the accompanying minerals and the formation and character of the pipes are fully discussed.

Section II., "The Matrix of the Diamond." This section occupies the greater part of the book; the various minerals occurring in the "blue clay" are very fully described; in fact, the material involves a mineralogical study of no mean order. Prof. Lewis deals with it very thoroughly, giving a complete list of the constituent minerals, and then describing each in order. The very pleasant manner in which the notes are given adds much to the interest of the subject. The occurrence is noted of a variety of garnet, called "demantoid" in microscopic crystals, so closely resembling diamonds in appearance as

to be sometimes mistaken for them. A very full account is given of the mica which is generally looked upon as an indication of the presence of diamonds.

As a result of his study of the diamantiferous clay, Prof. Lewis concludes that "There appears to be no named rock-type having at once the composition and structure of the Kimberley rock. For this reason, as also on account of its importance as the matrix of the diamond, it is now proposed to name the rock Kimberlite." At the conclusion of this section the resemblance between kimberlite and that class of meteorites known under the name of chondrites is pointed out.

Section III., "Kimberlite from the United States," only occupies a few pages, and consists of notes on various specimens of diamond-bearing rock that had been collected by Prof. Lewis, but which he evidently had not had the opportunity to thoroughly examine.

The book concludes with a short note by Prof. Bonney, giving data of the work done at the De Beer's mine up to date 1896.

CORRESPONDENCE.

ESTIMATION OF CARBON IN FERRO-CHROME.

To the Editor of the Chemical News.

SIR,—We are obliged to Prof. Arnold for his complimentary notice of our paper, and I beg to make the following observations on the objections he raises.

It will be noticed that the statement respecting Prof. Arnold's method is not an unqualified one. The kind of combustion furnace used (Bunsen's) and the approximate heat of the tube was given; after this, I was justified in stating the results. It is my opinion that the furnace used can be made as hot as those generally employed. In one particular, however, it did not comply with the conditions stated in Prof. Arnold's letter. The combustions were all made between 10 a.m. and 5 p.m. The inconvenience of waiting for night pressure during the summer months needs no comment. The gas-supply is through a 1½ (outside diam.) pipe, and gives as much gas as can be efficiently burned. It should be noticed in your correspondent's favour that it was necessary to coat the tubes with asbestos.

It was lamented in the article that so vague a description of the necessary tubes should be deemed sufficient; I am sorry to note that even now the Professor goes no further than to say "such tubes are not easy to obtain, but they are, nevertheless, obtainable." I have tried to get such tubes, and admit failure. It is a small thing to ask, will Prof. Arnold oblige myself and others by saying what the tubes are called and where they may be had?

As is surmised, I have never heated bright steel drillings in dry oxygen, but I am pretty certain of the difference between moist and dry oxygen. For instance, that even carbon cannot be combusted in chemically dry oxygen ("Combustion in Dried Gases," H. B. Baker, *Fourn. Chem. Soc.*, 1885; *CHEM. NEWS*, li., 150); but Prof. Arnold's oxygen is not chemically dry, and since traces of moisture make the most marked difference it is impossible to argue about the need for employing moistened asbestos with "almost chemically dry oxygen." Moreover, why the necessity of drying the gas so completely if it must needs be re-moistened. Our own arrangement, which is very efficient, is KHO solution and successive towers of KHO sticks and well-dried granular CaCl₂.

Water taken up in a current of gas cannot, of course, be deposited in a hotter place; but the diagram in "Steel Works Analysis" does not indicate, nor does the accompanying text insist, that the exit end should be the hotter. In fact, if the dimensions of the diagram be adhered to, the inlet tube is actually the

hotter—for the packing stops about equidistant from both ends—and in the general instructions for combustion it is expressly stipulated that the first plug (inlet end) shall be three burners within the furnace (p. 34), while the last plug is partly without the furnace (p. 36). It is right to state, though it by no means affects the instructions just noted, that the ends of our tubes were protected from direct flame radiation by a sheet of asbestos mill-board, and kept cool by means of a strip of filter paper resting with its loose ends in water.

The lead protoxide deposit was most noticeable in those cases which were combusted three and four hours.

The reason in favour of their non appearance is very "simple" indeed. Surely Prof. Arnold does not rely on the three asbestos plugs: they are all purposely perforated!—I am, &c.,

R. L. LEFFLER.

P.S.—It may be well to state plainly what is incidentally noted in the paper, that I alone am responsible for the remark on Prof. Arnold's method and what follows.—R. L.

The Laboratory,
Messrs. Thos. Firth and Sons, Lim.,
Sheffield, May 31, 1897.

ESTIMATION OF CARBON IN FERRO-CHROME.

To the Editor of the Chemical News.

SIR,—in March, 1896, having a number of ferro-chromes to test for carbon, I met with similar difficulties to those mentioned by Brearley and Leffler (CHEMICAL NEWS, vol. lxxv., p. 241), which led me to make experiments to find an efficient method.

These experiments took me in much the same direction as Brearley and Leffler, but ended, as I venture to think, in a method which, while giving good results, is not so complicated as theirs; the ordinary combustion furnace is used with a porcelain tube, the heat required being only a bright red. No difficulty being experienced from spurting.

I also found that with copper oxide alone only about half the carbon is obtained, but that by mixing copper oxide with litharge the whole of the carbon was oxidised in about twenty minutes.

The litharge was freshly-prepared by fusing red-lead—possibly, as suggested by Brearley, peroxide of lead might be better, if free from substances which might be given off and absorbed by the potash.

The Method.—The ferro-chrome is ground fine in an agate mortar. 0.5 grm. is then mixed with 4.5 grms. CuO and 1.5 grms. litharge, and the mixture placed in a porcelain boat. The boat is then placed in the furnace and the combustion proceeded with in the ordinary manner. In about twenty minutes the whole of the carbon is burnt off. The following results have been obtained by this method:—

11 per cent ferro-chrome	..	5.41 per cent carbon.
20 " "	..	5.67 " "
30 " "	..	7.13 " "
50 " "	..	7.94 " "

The object of using copper oxide with the litharge is to prevent the latter attacking the porcelain, which it does very rapidly when used by itself; it also prevents any spurting.—I am, &c.,

E. H. SANITER.

Messrs. Whittaker and Co. will publish immediately a work on Organic Chemical Manipulation, on which Dr. J. T. Hewitt, of the East London Technical College, has been engaged. The first part of the text-book will give an account of the methods adopted in organic analysis, and the determinations of molecular weight; the second part being devoted to a typical set of organic preparations, systematically arranged and intended to give an idea of the methods adopted in organic work.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 18, May 3, 1897.

Researches on the Composition of Wheats, and on their Analysis.—Aimé Girard.—Already inserted.

New Property of Kathodic Rays which reveals their Complex Constitution.—H. Deslandres.—This paper requires the three accompanying illustrations.

Partial Polarisation of the Radiations Emitted by some Sources of Light under the Influence of the Magnetic Field.—P. Egoroff and N. Georgiewsky.—The rays of hydrogen and of helium in Geissler tubes have hitherto not given any definite results. The metals employed (Cu, Tl, Zn, Ca, In, Mg, La, Ba, Fe) demonstrate polarisation.

Rôle of Peroxides in the Phenomena of Slow Oxidation.—A. Bach.—The author's experiments show that the theory of Hoppe-Seyler is devoid of foundation. They also show that the oxidation product of hydrogen contains an oxidiser more energetic than H_2O_2 , but showing with permanganate the same quantity of active oxygen. This oxidising agent is probably the tetroxide H_2O_4 , which is decomposed into $H_2O + O_2 + O$, and would act upon permanganate like H_2O_2 . What has been said concerning the formation and the oxidising action of the peroxides applies equally to the processes of oxidation which take place in the animal organism. The oxidising ferments which exist in the blood are probably nothing but these easily oxidisable substances eminently apt to form peroxides.

Study of the Action of Potassium Permanganate upon Cupric Bromide.—H. Baubigny and P. Rivals.—We have shown in a former paper that all the bromine of an alkaline bromide is set free if, after adding copper sulphate and permanganate to the solution we evaporate to dryness at an ordinary temperature, whilst the chlorides are not decomposed under the same conditions. They have ascribed this difference to the fact that the oxidising action of the permanganate is produced only upon the bromide. The permanganate therefore behaves with cupric bromide in a neutral liquid as it does with organic matter. In order that the total elimination of the bromine may be possible a large excess of copper must be present in a soluble state. In practice it is preferable to introduce a weight of permanganate notably in excess.

Constitution of Metallic Alloys.—Georges Charpy.—The author views alloys as either eutectic, those fusible at the minimum temperature, or definite. The existence of the latter class has been strongly contested, but the existence of the compounds Cu_3Sn and Cu_2Sb seems fully demonstrated. Among binary alloys there are two normal types of constitution. The first presents crystals of a pure body (which may be a single metal or a definite compound of two metals) inclosed in a second constituent, which is in general a eutectic mixture formed by the juxtaposition of two finely divided elements, one of which is that which forms the crystals. The second type is that of isomorphous mixtures formed of a single species of crystals occupying the entire mass. This second type is of very frequent occurrence.

Determination of Oxygen Dissolved in Sea-water.—Albert Levy and Felix Marboutin.

Compounds of Metallic Salts with Organic Bases.

—D. Tombeck.—It is known that metallic salts combining with ammonium chloride, either in the presence or the absence of water, forms compounds which have been studied by several chemists, especially by Isambert. The

author proposes the study of analogous bodies in which ammonia is replaced by the bases derived from ammonia.

A Compound of Silver Chloride and Monomethylamine.—R. IRRY.—The author having examined the ammoniacal silver chlorides extends his researches to compounds in which monomethylamine is substituted for ammoniacal gas.

Recognition of the Yellow of Naphthol S, and of Analogous Colouring-matters in White Wines and Liqueurs.—ALBERTO D'AGUIAR and W. da SILVA.—Already inserted.

MISCELLANEOUS.

Rheostats for enabling Street Currents to be used for Medical Purposes.—The idea of making use of large and powerful currents for operations requiring but weak currents is nothing new. M. Foveau de Courmelles has recently brought out a compact and convenient instrument for this purpose which can be readily fitted to a wall plug. It is specially adapted for use with continuous currents.

Carborundum Production and Use.—The Carborundum Company reports to the *Engineering and Mining Journal* that its works have produced during the year 1896, in round numbers, 1,191,000 pounds or 595½ tons of crystalline carborundum. Consideration at the present is given to the production in crystalline form only, but another important industry into which carbide of silicon promises to be a valuable adjunct will naturally increase the usefulness of the material. Some mention has been made of the experiments showing that carborundum can be used, and will, in all probability, take the place of ferro-silicon in the manufacture of steel. Professor Luhrmann, of Germany, recently wrote an article on this subject, indicating that in the use of carborundum there will be in Germany alone, approximately, 2500 tons consumed annually, provided its cost would not exceed 6 cents per pound. It may be used for this purpose in an amorphous form, and the Carborundum Company is prepared to furnish it at a price slightly under this figure. In the *Journal of the Franklin Institute* for February, 1897, will be found an excellent descriptive paper on the carborundum plant at Niagara Falls, from the pen of Mr. Fitzgerald, chemical engineer of the works. This industry stands as a conspicuous illustration of the possibilities of the electric furnace as the source of hitherto unknown and valuable products.

MEETINGS FOR THE WEEK.

MONDAY, 14th.—Society of Chemical Industry, 8. "Note on a Possible Danger from Fire involved in the Transport of Barium Peroxide in Wooden Barrels," by Dr. A. Dupré, F.R.S. "The Valuation of Commercial Nitrate of Soda," by Dr. Pauli. "Recent Improvements in Smokeless Compounds and in Processes of Manufacture," by Hudson Maxim. "Comparative Experiments on the Estimation of Phosphoric Acid," by A. Cameron. "The Strength of Commercial Formaldehyd Solutions," by W. A. Davis.

THURSDAY, 17th.—Chemical. 8. Ballot for the Election of Fellows. "Reduction of Perthiocyanoic Acid," by F. D. Chattaway, M.A., and H. P. Stevens, B.A. "Molecular Refraction of Dissolved Salts and Acids—Part II," by Dr. J. H. Gladstone, F.R.S., and W. Hibbert. "A Space Formula for Benzene," by Dr. J. Norman Collie, F.R.S. "The Production of some Nitro- and Amido-Oxy-picolines," by Dr. A. Lapworth and Dr. J. Norman Collie, F.R.S. "The so-called Hydrates of Isopropyl Alcohol," by Dr. T. E. Thorpe, F.R.S. "The Carbohydrates of the Cereal Straws," by C. F. Cross, E. J. Bevan, and C. Smith. "Further Experiments on the Absorption of Moisture by Deliquescent Substances," by H. Wilson Hake.

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VOL. LXXV., No. 1960.

FURTHER NOTE ON THE INFLUENCE OF A MAGNETIC FIELD ON RADIATION FREQUENCY.*

By Prof. OLIVER LODGE, F.R.S., assisted by Mr. BENJAMIN DAVIES.

REFERRING to a former communication of mine, on the subject of Zeeman's discovery, printed on page 513 of the *Proceedings of the Royal Society*, for Feb. 11 this year, vol. lx., No. 367, I wish to add an observation to those previously recorded, as I have recently acquired a concave Rowland grating ($3\frac{1}{2} \times 1\frac{1}{2}$ in. ruled surface, 14,438 lines to inch, 10 feet radius of curvature, being the one used by Mr. George Higgs), of which the spectra of the first and third orders on one side are very satisfactory.

It is said on page 513, "If the focussing is sharp enough to show a narrow, dark reversal line down the middle of each sodium line, that dark line completely disappears when the magnet is excited." With the greater optical power now available the dark reversal line is often by no means narrow, and, though in some positions of the flame it does still tend to disappear or become less manifest when the flame is subjected to a concentrated magnetic field, the reason of its partial disappearance is that it is partially reversed again—i.e., that a third bright line, as it were, makes its appearance in the midst of the dark line, giving a triple appearance to each sodium line.

More completely stated the phenomena are as follows:—After obtaining each sodium line with a prominently double aspect by manipulating the flame, the magnet is excited, and the dark band in the midst of each sodium line is then seen to widen out considerably in the region of most intense magnetisation, while a bright intrusion line makes its appearance. On closer examination this new line is seen to be double, by reason of a dark division down its middle; and I apprehend that with still more magnetic power this dark band might itself open out into two; but this last phenomenon I have not yet observed.

The whole sodium group is thus seen as if it were octuple. The effect is not due to a mere mechanical disturbance or re-arrangement of the gases of the flame by the agency of magnetism; because a nicol, placed in the rays emanating transversely to the magnetic lines of force, cuts off nearly all the visible magnetic effect when oriented so as to get rid of light whose plane of polarisation contains the lines of force—that is, of oscillations or revolutions whose electrical components are across or around the magnetic lines. That it does not cut off every trace of the effect appears to be due to the fact that the field of force is not strictly uniform, and so its lines are not strictly parallel.

The following is a summary of the different appearances that may be seen, according to the state of the flame and the strength of the field:—

At low temperature, and with the flame forward in the field, when each sodium line is sharp and single, magnetism widens it, and with a little more power doubles it, causing a distinct dark line down its middle. The same effect occurs with lithium and thallium lines.

At higher temperature, and with the flame partially behind the field, when each sodium line appears as a

broad hazy-edged double, magnetisation greatly widens the doubling, pushing asunder the bright components very markedly; stronger magnetisation reverses the middle of the widened dark band, giving a triple appearance; stronger magnetisation still reverses the middle once more, giving a quadruple appearance to the line. In every case a nicol, suitably placed, cuts off all the magnetic effect, and restores the original appearance of the line.

A curious circumstance is that although both lines, D_1 and D_2 , show the effect, D_1 , i.e., the less refrangible line, shows it best and most sharply. I should describe the effect on D_2 as a coarse widening of considerable amount, but without very clear definition; whereas the widening of D_1 , though perhaps no greater in amount, is decidedly better defined. There is no doubt but that, with my grating, D_1 is the line at which one finds oneself usually looking in order to see the details of the change best; and I can hardly suppose this to be subjective to the grating. I hope to show the effects at the *Soirée* of the Royal Society.

(The same thing is seen when salts of lithium or of thallium are introduced into the flame, and the components of the doubled red lines are more widely separated than the components of the doubled green lines, the effect being proportional to wave-length. The most interesting line to try was the red cadmium line, since this has been proved to be of specially simple constitution by Michelson. We have recently been able to get the cadmium spectrum well developed by means of a sort of spark arc between the magnet poles, maintained by an induction coil excited by an alternating machine; and we find that the magnetic doubling of the chief lines occurs in precisely the same way with the spark spectrum as with the flame spectrum, and that the red cadmium line behaves in the same way as the others. The magnetic effect is better seen, from a direction perpendicular to the line of force, when a nicol is interposed in the path of the light, but rotation of the nicol through 90° cuts it entirely off, accurately so when a small spark is the source of light.—May 31).

SANITARY PROBLEMS CONNECTED WITH MUNICIPAL WATER-SUPPLY.*

By Prof. W. P. MASON.

THIS subject is in some danger of being over-written to-day; but we have about us material evidence that it is not over-studied, especially by those Boards of public officials whose responsibilities are often much greater than their knowledge of sanitary principles. The public also is far from being well posted on the matter, and one encounters all sorts of odd views, which are remarkable not only for their character, but also for the tenacity with which they are held.

Ocular evidence of purity is quite sufficient for most people. The bright and limpid water from a well which drains a grave-yard is counted a blessing by those who would shudder at the thought of a cholera-ship touching at one of our most distant ports. Nor is faith in the self-purifying power of running streams any less pronounced. The author had the following curious criticism made on his report condemning the use of a sewage-laden river-water:—"We would hint to Prof. Mason that every impurity which enters the river is either heavier or lighter than water. If it be heavier, it sinks quietly to the bottom; if it be lighter, it will remain on the surface a few hours, when it will be blown ashore by the wind."

"Water taken midway between the surface and bottom of the river will always be found as pure as the best spring water."

* A Paper read before the Royal Society, June 3, 1897.

* Abridged from a Lecture delivered before the Franklin Institute March 19th, 1897.

So long as such notions find expression in the daily press, so long, we may be sure, are the people ignorant and misinformed upon questions very nearly touching their safety, and so long there is direct need of suitable sanitary education.

To the writer's way of thinking, a land should be looked upon as watered by its smaller lakes, its springs, and its brooks and streams, and sewerage by its great—especially its navigable—rivers. Its water sources should be protected by law with exceeding care, and no stream or river should be added to its list of drains, except after proper consideration by the State Board of Health, followed by legislative permission.

Cases such as the typhoid fever outbreak at Plymouth, Pa., impress upon us the necessity of caring for our watersheds and the ultimate ramifications of the tributaries to our sources of supply.

Many readers are probably familiar with the excellent experiments carried on at Lawrence by the Massachusetts State Board of Health, which go to show that, by carefully conducted intermittent filtration through beds of sand or gravel stones, city sewage may be converted into what may practically be called potable water. Let these two very important points be always kept in mind however:—First, the filtration must be intermittent, for if it be continuous the atmospheric oxygen necessary to the activity of the purifying organisms of nitrification becomes excluded, and purification ceases. Secondly, the "dose" of applied sewage must not be larger than that quantity which experiment has shown to be capable of disposal by the filter.

The general trend of our information goes to establish the fact that proper care of the watershed is as necessary as it is unusual, and I firmly believe that such care should be carried even to the extent of protecting the groundwater for a reasonable distance before it enters the draining brooks of the district.

After a suitable and well-protected gathering ground has been secured, and after the water has been started on its way to the consumer, other opportunities for pollution not infrequently arise. Open channel-ways, as a means of conveying water to a town, are quite commonly seen, and care is not always taken that pollution shall not reach the water during its flow therein. We find a very noteworthy case of contamination, under just such circumstances, recorded in the history of the cholera epidemic at Messina, Sicily, in 1887. The plague lasted from the 10th of September to the 25th of October, during which time there were some 5000 cases and 2200 deaths. The Government felt that a very possible cause for the rapid spread of the scourge lay in a contaminated drinking water, and enquiry fully confirmed this suspicion. The water as it left the gathering grounds was of excellent quality, but, for the benefit of the Messina washerwomen, a portion of the water was deflected before reaching the wells, and turned into neighbouring washing-pools of stone. A fair proportion of this deflected water, after having been used for laundry purposes, found its way back into the open channel, and continued its course to the city. Further contamination occurred within the town itself; the water-mains and sewers were of unglazed tiles, very leaky, and the sewers were at times found on the top of, and parallel with, the water-mains themselves. The Government sent tank ships filled with pure "Serino" water, supplied the people therewith, and the daily number of cases immediately fell from seventy to five.

Keeping our attention upon typhoid fever, it will be remembered that two conceptions of its origin are entertained by opposing schools of bacteriologists. On the one hand, it is held that the typhoid germ is always the offspring of a bacillus of its own kind; while, on the other side, there are those who believe—and it is a very conceivable belief—that the progenitor in question is often a saprophyte which takes on its pathogenic properties by cultivation through successive generations under favourable conditions.

Many illustrations are available, in the world of larger vegetation, of greater changes in structure due to cultivation under an altered environment.

Roux and Rodet are perhaps the leaders among those who claim a saprophytic ancestry for the typhoid germ, but they are not without a strong following in this country.

Whatever may be the final decision of the specialists upon this knotty point, it is our manifest duty to adopt for the present the saprophyte theory as our working formula, and to protect water-supplies from the infiltration of animal waste material.

Directly connected with the conveying of water in open channels comes the question of the self-purification of water under such circumstances. Agitation and aëration do certainly aid in preventing abundant growth of Algæ, and an undoubted improvement in the quality of water results from the establishment of a fountain in a too quiet reservoir; but the expectations of those who hope to thus easily eliminate pollution of a serious character will not be realised.

Sedimentation plays a part in the general purification during open flow, but it is commonly a small one, particularly small in such streams as stand in special favour with the public, because of the riffles and other interruptions in their courses.

Sedimentation, when we consider the question of drawing our supply from a lake, particularly a large one, becomes of prime importance, and, as the element of time enters largely into the consideration of these cases, material changes for the better are often noticeable in lake waters; thus the tributary of a lake may be undesirable for domestic use, while its outlet may be entirely satisfactory.

Referring to the suggestion recently published by one of our sanitary engineers, to the effect that the cleaning of storage reservoirs is all a mistake, and that it would be far better practice to leave the vegetable *débris* where Nature placed it, it must be replied that comparative experiments upon such reservoirs have shown that improved water unquestionably follows cleaning. No stripping of the soil from the bottom of the Vyrnwy reservoir, which supplies Liverpool, was done, but the water is filtered before delivery for consumption.

Filtration is so common in Europe that the same care in storage is not so necessary as in the United States, where the practice is to supply the raw water direct to the consumer.

Waters from underground sources should be distributed for use as soon as possible after they have been brought to the surface, for they are commonly well supplied with plant-food in solution, and, under the influence of light and air, there is danger of abundant development of objectionable Algæ if much time for open storage be allowed. With surface-waters the case is quite the reverse, and long storage becomes a distinct advantage, if the reservoir be clean. Bacteria often die but slowly, and, although a large percentage of their number will disappear through storage, it should not be forgotten that they are very small and very light, and consequently are very long in settling, so that it should not be expected that a reservoir could do the efficient work accomplished by a filter.

From whatever source the water may be derived, it is the common American practice to deliver it "raw" to the consumer, even when its appearance is distinctly unsightly. Such, however, is not the European custom. Public sentiment abroad demands that surface-waters should receive efficient purification before they are distributed for domestic use. As a result, filters are established, or arrangements are contemplated for their erection, to filter waters, of a degree of natural purity, equal to the best supplies America can show. We on this side of the Atlantic would consider the expenditure of money for the purpose of purifying such waters as we find at Liverpool and Zurich quite unnecessary and superfluous; Europeans think differently, however, and their notions are best.

expressed by Voltaire's apothegm "Le superflu, chose très nécessaire."

It is very amusing to note the care with which Americans—perhaps from Albany, Pittsburg, or Chicago—scrutinise the water offered them in foreign capitals, when what they are in the habit of drinking at home would not be tolerated for an instant in the great cities of Europe.

The day is past when we could feel a sense of superiority over the crowded millions of the old world, because of the relative magnitude and consequent initial purity of the sources of our water-supplies. Europe has, of late years, expended much labour and capital in substantial plants, that make for sanitary betterments, while we have continued upon a conservative course, forgetful that our populations and industries have been growing, and that the rivers our fathers drank from with pleasure and safety have become charged with the refuse of up-stream communities, and converted into what may be properly styled the county sewers.

There is no system of filtration so expensive but that a community can well afford to introduce it rather than to drink a dangerous water in its raw state, and this, too, from purely economic considerations, and leaving out of sight all ethical questions whatsoever.

THE VISCOSITY OF MERCURY VAPOUR.*

By A. A. NOYES, Ph.D., and H. M. GOODWIN, Ph.D.

THE uncertainty which attaches to the specific heat ratio of gases as a means of distinguishing between monatomic and polyatomic molecules has been recently made evident by the extended discussions of the significance of that property in connection with the atomic weights of argon and helium. It is therefore of great interest to investigate other properties which may be expected to be related to the atomicity of the molecule. The authors have therefore undertaken the investigation of one of these, viz., the viscosity, or internal friction, in order to determine if any marked difference in its value exists in the case of gases with monatomic and those with polyatomic molecules. To this end they have made comparative measurements of the viscosity of hydrogen, carbon dioxide, and mercury vapour, at the boiling temperature of the latter. The authors thought that monatomic molecules might prove to be much smaller than polyatomic ones, since it seems *a priori* not improbable that the spaces between the atoms of the latter are large in comparison with the atoms themselves. The experiments described show, however, that no marked distinction exists between monatomic and polyatomic gases in this respect.

The method used by them in determining the relative viscosity consisted in measuring the quantities of the different gases which, under a constant difference of pressure, passed in a given time through the same capillary kept at a definite constant temperature. The apparatus and experimental method employed were, for given reasons, quite different from the usual ones, and are briefly described. The capillary used in the most complete series of experiments consisted of a glass tube 74 c.m. long and 0.34 m.m. internal diameter (a smaller capillary, 49 c.m. long and 0.22 m.m. in diameter, was used in a preliminary series); it was compactly bent up on itself so as to form five vertical segments with, of course, four elbows, the beginning and the end of the capillary part of the tube being at the same level. To these ends were fused pieces of ordinary glass tube, one of which was bent horizontal, and provided at the point with a ground joint; to the other was fixed a vertical T-piece. This was all placed in a heavy steel cylinder, 30 c.m. high and 2.8 c.m. internal diameter, having a small orifice at the side through which the ground joint

protruded for about 1 c.m., the whole being held in position by loosely-packed asbestos. Although the capillary was vertical, the influence of gravity was eliminated by reason of the fact that the ascending and descending parts were of equal length. The top of the cylinder was closed by an iron plate screwed down by a nut, both plate and nut were perforated through the centre, and into the latter was welded an iron tube, projecting vertically, 25 c.m. in length and 2.5 c.m. diameter. The cylinder was covered, except on the bottom, with a jacket of asbestos about 5 c.m. thick, and spirals of copper wire were wound round the vertical tube, which was to serve as a condenser, in order to increase the cooling surface. Pure mercury was placed in the cylinder and boiled vigorously by means of a flame underneath. The capillary was thus kept at the temperature of the boiling mercury at atmospheric pressure. No regard was paid to the variations of temperature arising from changes in barometric pressure, as their effect would evidently be entirely negligible.

Any desired difference of pressure at the two ends of the capillary was obtained by inserting a tube in the ground joint and connecting it with a large air reservoir, which was in turn connected with a suction-pump and furnished with an open mercury manometer. The gas, or vapour entered at the other end of the capillary, always under atmospheric pressure.

In making the experiments the rate of flow of the mercury vapour was first determined in the following manner:—While the cylinder was being heated carbon dioxide was forced through the capillary to prevent the condensation in it of liquid mercury and the formation of its oxide. After the mercury was boiling actively and its vapour entirely enveloped the capillary, shown by a thermometer inserted into the vertical iron tube, it was connected with the suction pump and mercury vapour drawn through for half an hour. The carefully ground end of a weighed bulb was then inserted in the ground joint, and its other end connected by means of a clamped rubber tube with the air reservoir, in which the desired reduction of pressure had been produced. At a definite moment the clamp was opened and the time noted. The mercury vapour was found to be completely condensed in the weighed tube, about 2 or 3 c.m. from the ground joint. There was a slight and unavoidable leakage* through the ground joint, and it was therefore necessary to re-adjust the pressure occasionally. It could easily be kept constant to 0.2 or 0.3 m.m. Usually after sixty minutes the clamp was closed, and at a noted instant the bulb was removed and subsequently weighed. The capillary was removed from the cylinder and the lower end of the T-piece closed by fusion. A glass tube, long enough to project above the end of the iron tube, was then fused to the upper part of the T-piece, and the capillary was then ready for the experiments with carbon dioxide and hydrogen. It was replaced in the cylinder as before, and the projecting vertical glass tube connected through suitable wash-bottles with the gas generator.

In order to maintain the gas entering the capillary at atmospheric pressure, a T-tube was inserted between the wash-bottles and the capillary, and its perpendicular arm was turned downwards and caused to dip into sulphuric acid, barely below its surface. The cock of the generator was opened sufficiently to cause the gas to bubble out steadily through the sulphuric acid.

The transpiration measurements were made as in the case of the mercury. The carbon dioxide flowing through in a definite time, was determined by absorption in weighed tubes filled with lumps of soda-lime. The hydrogen was burnt by passing it over hot copper oxide contained in combustion-tubes from which the air was previously displaced by carbon dioxide, and the water collected in weighed calcium chloride tubes.

* In the case of the mercury experiments no error could arise from this source, as the leakage was inward. In the case of those of carbon dioxide and hydrogen it was proved by blank experiments that the amounts of carbon dioxide and water which leaked in were less than 1 per cent of the total weight.

The results are given in the following tables. The first column gives the symbol of the substance; the second, the atmospheric pressure, p_1 ; the third, the difference in pressure ($p_1 - p_2$); the fourth, the time, t , expressed in hours; the fifth, the weight, w , in grms. of the substance weighed; the sixth, the mean weight transpired in one hour as computed from the separate check experiments; and the last, the quotient obtained by dividing this weight by the molecular weight, m , of the substance, the time, and the pressure function ($p_1^2 - p_2^2$)*.

Series I.—Small Capillary.

	p_1	$p_1 - p_2$	t	w	$w : t$	$\frac{w \times 1010}{t m(p_1^2 - p_2^2)}$
Hg..	753	200	1½	0.738	—	—
	753	—	1½	0.740	—	—
	756	—	1½	0.745	0.494	93.6
	755	300	1	0.685	—	—
	755	—	1	0.686	0.686	93.6
	754	400	1	0.834	—	—
	754	—	1	0.831	0.833	92.9
	752	200	1	0.237	—	—
	752	—	1	0.239	—	—
	752	—	1½	0.357	0.238	205
CO ₂ .	759	300	1	0.329	—	—
	759	—	1	0.327	—	—
	766	—	1	0.324	0.327	203
	766	400	1	0.400	—	—
	766	—	1	0.396	—	—
	759	—	1	0.396	0.397	202
	766	300	1	0.267	—	—
	765	—	1	0.264	0.265	779
	766	150	1	0.557	—	—
	766	—	1	0.557	0.557	1517
H ₂ ..	766	300	1	1.009	—	—
	766	—	1	1.006	1.008	1496
	766	300	1	2.728	—	—
	766	—	1	2.746	—	—
Hg..	764	—	1	2.743	2.739	371

Series II.—Large Capillary.

	p_1	$p_1 - p_2$	t	w	$w : t$	$\frac{w \times 1010}{t m(p_1^2 - p_2^2)}$
Hg..	769	150	1	1.548	—	—
	769	—	1	1.548	1.548	374
	769	300	1	2.763	—	—
	769	—	1	2.764	—	—
CO ₂ .	769	—	1	2.739	2.755	373
	756	150	1	0.704	—	—
	765	—	1	0.706	0.705	774
	765	300	1	1.267	—	—
H ₂ ..	765	—	1	1.264	1.265	779
	766	150	1	0.557	—	—
	766	—	1	0.557	0.557	1517
	766	300	1	1.009	—	—
Hg..	766	—	1	1.006	1.008	1496
	766	300	1	2.728	—	—
	766	—	1	2.746	—	—
	764	—	1	2.743	2.739	371

The agreement of the values of the last column in the cases of the same substances under different pressures show that the capillaries are of sufficient length and small enough bore to give the true values of the viscosity coefficients.

The relative viscosity coefficients of the different gases were calculated from the values of the last column in the tables, and gave the following results:—

$$\frac{\eta \text{ Hg}}{\eta \text{ CO}_2} = 2.17 \dots \dots \dots (\text{First series}).$$

$$\frac{\eta \text{ Hg}}{\eta \text{ CO}_2} = 2.08 \cdot \frac{\eta \text{ Hg}}{\eta \text{ H}_2} = 4.04 \cdot \frac{\eta \text{ CO}_2}{\eta \text{ H}_2} = 1.94 (\text{Second series}).$$

* In the calculation of this quantity, the same mean value of p_1 was used in all the experiments of each series, namely, 760 for those with the smaller capillary, 765 for those with the larger.

The relative values for mercury and carbon dioxide agree within about 4 per cent.

The corresponding values of the mean cross-sections as calculated are—

$$\frac{q \text{ Hg}}{q \text{ CO}_2} = 1.02 \quad \cdot \quad \frac{q \text{ Hg}}{q \text{ H}_2} = 2.48;$$

that is to say, the average cross-section of the mercury molecule or atom is very nearly the same as that of the carbon dioxide molecule, and is about two and a half times as large as that of the hydrogen molecule.

These results indicate that atoms and molecules are of the same order of magnitude, and that the spaces between the atoms within the molecule, if any exist, are not large in comparison with those occupied by the atoms themselves; and, consequently, the viscosity of gases, or any other property which, like it, is dependent only on the size or form of the molecules, is not adapted for distinguishing between monatomic and polyatomic molecules.

The mercury and carbon dioxide molecules have, as we have seen, the same cross-section, and therefore, assuming both to be of the same general form, they occupy the same volume. The mass of the former is, however, 4.55 times as great as that of the latter. The density of the mercury molecule is consequently greater in this same proportion. But this difference is not marked enough to make it necessary to attribute it to free spaces within the carbon dioxide molecule.

ON THE PURIFICATION OF CERIUM.

By MM. WYROUBOFF and A. VERNEUIL.

In spite of the large number of researches on cerium and its compounds there still exists much uncertainty with regard to its most characteristic chemical properties.

These uncertainties come from two causes—the absence of any method enabling us to easily obtain cerium quite free from its accompanying earths, and the inadequacy of the methods used for determining its atomic weight. These two voids keep us in a circle of errors, from which we have no escape, for we recognise the purity of a body by its atomic weight, and we are fixing the atomic weight in a product we cannot recognise as pure.

Among the numerous methods for the separation of cerium, there are only two that are held in general favour—the treatment by chlorine in the presence of alkalis employed by Mosander, and fusion with nitre proposed by Debray. Both have the same fault; they are not based on any precise reaction, and exist under the most disadvantageous conditions with regard to the separation in view.

Cerium is distinguished from all similar metals by a very characteristic property,—the existence of a very stable higher oxide, capable of easily forming basic salts which are generally insoluble. It is therefore very natural to take advantage of this property to isolate the metals of its group, or of neighbouring groups. This is, in fact, what has been attempted by Mosander and Debray; but they were not aware of the existence, between the lower oxide, CeO₂,* and the higher oxide, Ce₂O₄, of an intermediate oxide, Ce₆O₇ = Ce₃O₄.3CeO, very stable, and also giving insoluble basic salts, depending in most cases on the oxidation of the CeO and the reduction of the Ce₂O₄. This oxide becomes even more stable when the cerium is in the presence of lanthanum and didymium, both more basic than itself, for it then invariably forms a complex oxide, Ce₂O₄.3MO, in which M = Ce + La + Di in proportions varying according to circumstances. Now, in Mosander's method, even the prolonged action of chlorine

* We consider cerium as being bivalent in its lower oxide, and we shall give, before long, the reasons which have forced us to adopt this old opinion of Berzelius.

will hardly bring about the formation of this intermediate oxide; and in Debray's process, the very first thing caused by the action of heat is precisely its production. It is for this reason that, in order to produce a body, even the purity of which is never certain, it is necessary to repeat the operation many times.

The existence of the intermediate oxide once admitted, the problem of the separation of cerium is much simplified, for it is reduced to the two following points:—we can either prevent the formation of this oxide, or else find out under what conditions it will separate into its two component parts, Ce_2O_3 , forming insoluble basic salts, and $\text{CeO} + \text{LaO} + \text{DiO}$, giving neutral soluble salts. It is this latter, and far simpler, solution of the difficulty that we have endeavoured to find.

The oxides resulting from the moderate calcination of the oxalates are dissolved in warm nitric acid. There results a partial reduction, a disengagement of oxygen, and the formation of the intermediate oxide. The solution is evaporated to the consistency of a syrup, to drive off the excess of acid. The mass is then easily dissolved in water, forming a limpid yellowish solution, which should be diluted to about 4 per cent of oxide. If to this warm solution we add 5 per cent of nitrate of ammonia the intermediate oxide is completely dissociated; the whole of the oxide Ce_2O_3 is precipitated as a basic salt, $(\text{Ce}_2\text{O}_3)_4\text{N}_2\text{O}_5$, and the protoxides remain in solution, which takes the violet tint of didymium salts. The precipitate deposited is washed with 5 per cent nitrate of ammonia; it contains cerium as free from didymium and lanthanum as from the yttria earths. These earths cannot be associated with cerium except when the latter is in the state of the oxide Ce_2O_3 , 3CeO , and the nitrate of ammonia—by making the salt of Ce_2O_3 insoluble—renders the existence of this oxide impossible. It is true that by this method we only get about 75 per cent of the cerium present, but there is nothing to prevent us from repeating the operation by precipitating the filtrate by oxalic acid, calcining the oxalates, and re-dissolving in nitric acid.

In cases when the mixture of oxides contains more than 50 per cent of cerium, it is no longer integrally soluble in nitric acid. It is then necessary to dissolve the oxalates in nitric acid, and add peroxide of hydrogen and ammonia. This is boiled, to transform the brown peroxide formed into yellow ceroso-ceric hydroxide; this hydroxide, after washing, is dissolved in nitric acid, and the operation continued as we have just described. The cerium thus obtained is not yet, however, pure; it contains all the thorina which was present in the mixture of the oxides.

We would further remark, however, that the thorina exists only in the 75 per cent of cerium of the first operation. If therefore we wish to obtain, without further purification, cerium completely free from thorina, it suffices to repeat the operation on the oxides remaining in solution after the first precipitation. In any case it is easy to get rid of the thorina, by treating the oxalates, or better still the nitrates, made as near as possible neutral, by a concentrated solution of carbonate of ammonia, to which is added a little caustic ammonia. The thorina is easily dissolved, at the same time as a small portion of the other earths; after two exhaustions there is not more than 1 per cent of thorina left. To remove this last trace we crystallise the sulphate at 60° in a liquor entirely free from free sulphuric acid; the thorina accumulates in the mother-liquor, forming with cerium a very soluble double sulphate.

There remains now nothing but to rid the cerium of iron, which it retains with great persistence. To do this it is necessary to precipitate it several times from its chloride and nitrate solutions, by means of oxalic acid, in hot and acid solution; we can also eliminate the sulphate by dehydration at a high temperature— 400° to 450° ; the iron then remains in an insoluble state.

Cerium thus purified is always identical in composition,

and its atomic weight does not vary, as we shall show in a future note. Its oxide, calcined at a high temperature, is absolutely white when cold. All yellow, chamois, or rose tints indicate the presence of impurities, which can always be eliminated by known methods (*Laboratoire de Chimie du Museum d'Histoire Naturelle*).

M. MOISSAN added the following remarks *à propos* of the communication of MM. Wyruboff and Verneuil "On the Preparation of Oxide of Cerium."

The important researches of MM. Wyruboff and Verneuil induce me to mention the process I adopted to obtain the perfectly white oxide of cerium, which I used in the preparation of carbide of cerium.

I started with an oxide of cerium no longer giving an absorption spectrum in a concentrated solution. I transformed it into carbide in the electric furnace. By dissolving this in cold water, either pure or slightly acidulated, I obtained a complex mixture of carbides of hydrogen, the composition of which would not remain constant during the whole length of the reaction.

Three hundred grms. of this carbide, finely powdered, was then treated with a solution of nitric acid, kept very dilute in order to limit the action. The carbide remaining was taken up in a fresh quantity of weak acid, but the solution was not allowed to be complete. The solution obtained by this second treatment furnished, by simple calcination, absolutely white oxide of cerium.

The iron was found in the first liquid, and the thorina in the residue of unattacked carbide.

This method of preparation gave me a white oxide of cerium, while that from the first solution was of a rose-colour, and that from the residue of a yellowish green tint.—*Comptes Rendus*, vol. cxxiv., No. 22, p. 1230.

FOURTH ANNUAL REPORT OF COMMITTEE ON ATOMIC WEIGHTS.

RESULTS PUBLISHED IN 1896.*

By F. W. CLARKE.

(Concluded from p. 283).

Nitrogen.—Among the ratios measured by Penny and by Stas relative to the atomic weights of nitrogen, those connecting the chlorides and nitrates of potassium and sodium were highly important. These are now re-determined by Hibbs (Doctoral Dissertation, University of Pennsylvania, 1896, by J. G. Hibbs, *Journ. Am. Chem. Soc.*, xviii., 1044) in a different way. The nitrates were heated in gaseous hydrochloric acid, and so converted easily into chlorides, with considerable accuracy. The data are as follows with vacuum weights, and reduced with $\text{O} = 16$, $\text{K} = 39.11$, $\text{Na} = 23.05$, and $\text{Cl} = 35.45$.

Weight KNO_3 .	Weight KCl .	Atomic wt. N.
0.11090	0.08177	14.011
0.14871	0.10965	14.010
0.21067	0.15523	14.013
0.23360	0.17223	14.011
0.24284	0.17903	14.014
	Mean ..	14.0118
Weight NaNO_3 .	Weight NaCl .	Atomic wt. N.
0.01550	0.01066	14.011
0.20976	0.14426	14.011
0.26229	0.18038	14.014
0.66645	0.45829	14.014
0.93718	0.64456	14.008
	Mean ..	14.0116

* *Journal of the American Chemical Society*, xix., No. 5.

These results seem to be exceedingly good, and the process has the advantage of great simplicity. The work was done under the direction of Prof. E. F. Smith.

Arsenic.—In the dissertation already cited Hibbs gives some determinations of the atomic weight of arsenic. Sodium pyroarsenate was heated in gaseous hydrochloric acid and so converted into chloride. The latter was perfectly white, unfused, and showed no trace of arsenic. I subjoin the vacuum weights, and the values found for arsenic when $O=16$, $Na=23.05$, and $Cl=35.45$.

Weight $Na_3As_2O_7$.	Weight $NaCl$.	Atomic wt. As .
0.02177	0.01439	74.904
0.04713	0.03115	74.921
0.05795	0.03830	74.927
0.40801	0.26981	74.901
0.50466	0.33345	74.916
0.77538	0.51249	74.917
0.82897	0.54791	74.917
1.19124	0.78731	74.926
1.67545	1.10732	74.928
3.22637	2.13267	74.901
Mean		74.9158

Magnesium.—Atomic weight determined by Richards and Parker (*Zeit. Anorg. Chem.*, xiii., 81), who studied the carefully purified chloride. First, a gravimetric series, with all weights reduced to a vacuum.

Weight $MgCl_2$.	Weight $AgCl$.	Atomic wt. Mg .
1.33550	4.01952	24.368
1.51601	4.56369	24.350
1.32413	3.98528	24.369
1.40664	4.23297	24.386
1.25487	3.77670	24.373
Mean		24.369

The remaining series of experiments are of the usual volumetric character.

Second Series.

Weight $MgCl_2$.	Weight Ag .	Atomic wt. Mg .
2.78284	6.30284	24.395
2.29360	5.19560	24.379
2.36579	5.35989	24.366
Mean		24.380

To this series the authors attach less importance than to the others.

Third Series.

Weight $MgCl_2$.	Weight Ag .	Atomic wt. Mg .
1.99276	4.51554	24.349
1.78870	4.05256	24.363
2.12832	4.82174	24.268
2.51483	5.69714	24.372
2.40672	5.45294	24.369
1.95005	4.41747	24.377
Mean		24.365

Fourth Series.

Weight $MgCl_2$.	Weight Ag .	Atomic wt. Mg .
2.03402	4.60855	24.360
1.91048	4.32841	24.364
2.09932	4.75635	24.362
1.82041	4.12447	24.362
1.92065	4.35151	24.363
1.11172	2.51876	24.363
Mean		24.362

These values are computed with $O=16$.

When $O=15.88$, $Mg=24.179$. The last series outweighs all the others.

Cadmium.—Hardin's determinations of the atomic weight of cadmium resemble those which he made upon silver. First, the chloride, in solution with potassium cyanide, was electrolysed in a platinum dish. The weights in this and the other series are all reduced to a vacuum. Computations made with $Cl=35.45$ and $O=16$. Data as follows:—

Weight $CdCl_2$.	Weight Cd .	Atomic wt. Cd .
0.43140	0.26422	112.054
0.49165	0.30112	112.052
0.71752	0.43942	112.028
0.72188	0.44208	112.021
0.77264	0.47319	112.036
0.81224	0.49742	112.023
0.90022	0.55135	112.041
1.02072	0.62505	112.002
1.26322	0.77365	112.041
1.52344	0.93315	112.078
Mean		112.038

Secondly, the bromide was treated in the same way. The data were reduced with $Br=79.95$.

Weight $CdBr_2$.	Weight Br .	Atomic wt. Cd .
0.57745	0.23790	112.031
0.76412	0.31484	112.052
0.91835	0.37842	112.067
1.01460	0.41808	112.068
1.15074	0.47414	112.053
1.24751	0.51392	112.019
1.25951	0.51905	112.087
1.51805	0.62556	112.076
1.63543	0.67378	112.034
2.15342	0.88722	112.041
Mean		112.053

In a third series of experiments cadmium was thrown down simultaneously with silver in the same electric current. Weights and results as follows, with $Ag=107.92$.

Weight Ag .	Weight Cd .	Atomic wt. Cd .
0.24335	0.12624	111.928
0.21262	0.11052	111.991
0.24515	0.12720	111.952
0.24331	0.12616	111.916
0.42520	0.22058	111.971
Mean		111.952

Mean of all the twenty-five experiments, $Cd=112.027$.

Mercury.—Atomic weight also determined electrolytically by Hardin, in the same paper with his work upon silver and cadmium. With the oxide he obtained unsatisfactory results; but with the chloride, bromide, and cyanide he did better. With the chloride, when $Cl=35.45$, his data, with vacuum weights, are as follows:—

Weight $HgCl_2$.	Weight Hg .	Atomic wt. Hg .
0.45932	0.33912	200.030
0.54735	0.40415	200.099
0.56002	0.41348	200.053
0.63586	0.46941	199.947
0.64365	0.47521	200.026
0.73281	0.54101	199.988
0.86467	0.63840	200.038
1.06776	0.78825	199.946
1.07945	0.79685	199.912
1.51402	1.11780	200.028
Mean		200.006

With the bromide, when $Br=79.95$, Hardin found these weights and values:—

Weight HBr ₃ .	Weight H ₂ .	Atomic wt. H ₂ .
0'70002	0'38892	199'898
0'56430	0'31350	199'876
0'57142	0'31750	199'938
0'77285	0'42932	199'832
0'80930	0'44955	199'814
0'85342	0'47416	190'911
1'11076	0'61708	199'869
1'17270	0'65145	199'840
1'26186	0'70107	199'899
1'40142	0'77870	199'952
Mean		199'883

With the cyanide, when C = 12'01 and N = 14'04, Hardin found—

Weight HgC ₂ N ₂ .	Weight Hg.	Atomic wt. Hg.
0'55776	0'44252	200'063
0'63290	0'50215	200'092
0'70652	0'56053	200'038
0'80241	0'63663	200'075
0'65706	0'52130	200'057
0'81678	0'64805	200'103
1'07628	0'85392	200'077
1'22615	0'97282	200'071
1'66225	1'31880	200'057
2'11170	0'67541	200'077
Mean		200'071

Finally, Hardin made use of Faraday's law, throwing down mercury and silver simultaneously in the same electric current. The equivalent weights are as follows, reduced with Ag = 107'92 :—

Weight Hg.	Weight Ag.	Atomic wt. Ag.
0'06126	0'06610	200'036
0'06190	0'06680	200'007
0'07814	0'08432	200'021
0'10361	0'11181	200'011
0'15201	0'16402	200'061
0'26806	0'28940	199'924
0'82808	0'89388	199'929
Mean		199'996

The general mean of all four series is—

Hg = 199'989.

Tellurium.—In all determinations hitherto made of the atomic weight of tellurium, the material has been derived from metallic tellurides. Chikashige (*Journ. Chem. Soc.*, lxi., 881) now gives a series of experiments upon tellurium obtained from Japanese native sulphur, using Brauner's method. The tetrabromide was titrated with solutions of silver, and the following data were obtained. Computations were made upon the basis of O = 16.

Weight TeBr ₄ .	Weight Ag.	Atomic wt. Te.
4'7812	4'0348	127'57
4'3059	4'1547	127'61
4'5929	4'4314	127'58
Mean		127'587

Tungsten.—Schneider (*Journ. Prakt. Chem.*, [2], liii., 288) objects to the determinations published by Pennington and Smith, regarding them as too high. He attributes their highness to the fact that very small quantities of material were handled, and thinks that there may have been mechanical losses of small particles during the long heating of the substance weighed. He now gives new determinations of his own, with tungstic oxide carefully freed from molybdenum, and made partly by reduction of the oxide, partly by oxidation of the metal. Results as follows, with the percentage of tungsten in tungsten trioxide stated in a third column :—

2'0728 grms. WO ₃ gave	1'6450 W	79'323
4'0853	" " "	79'309
6'1547	" " "	79'307
1'5253	" W "	79'311
3'1938	" " "	79'304
3'7468	" " "	79'314
Mean		79'311

Hence with O = 16, W = 184'007.

On the other hand, Shinn (Doctoral Thesis, University of Pennsylvania, 1896), working in Smith's laboratory, obtains some data corroborative of Pennington and Smith. In this series tungsten was oxidised to tungsten trioxide. Results as follows, computed with O = 16 :—

0'22297 grm. W gave	0'28090 grm. WO ₃	184'720
0'17200	" " "	184'964
0'10989	" " "	184'753
0'10005	" " "	185'206
Mean		184'910

The cause of the difference between the values found and those of Schneider is yet to be made out.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, June 3rd, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

MESSRS. Thomas Tickle and Thomas Girtin were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. John Ball, Ph.D., 18, Redshaw Street, Derby; Alec Alfred Beadle, Beadonwell, Belvedere, Kent; James Walter Horseman, 5, South Parade, Chelsea, S.W.; Charles John Jodrell Mansford, B.A., Lady Manners Grammar School, Bakewell, Notts.; Thomas Southern, jun., 2, Cherry Mount, The Cliff, Higher Broughton, Manchester; Francis Samuel Young, M.A., Mill Hill School, N.W.

Of the following papers those marked * were read :—

*67. "On the Thermal Phenomena attending the Change of Rotatory Power of Freshly-prepared Solutions of certain Carbohydrates; with some Remarks on the Cause of Multirotation." By HORACE T. BROWN, F.R.S., and SPENCER PICKERING, F.R.S.

During an investigation of the thermal changes attending hydrolysis under enzyme action, whose results are described in the next paper, it became necessary to enquire whether the change in the multi-rotation of certain sugars is attended with any heat disturbance, as it is now well-known that, at any rate, dextrose and maltose are liberated by hydrolysis in the "birotatory" state. The authors find that the changes of rotation experienced by dextrose, lævulose, and milk-sugar in passing from the optically unstable α - to the optically stable β -form, are accompanied by distinct thermal effects which, although taking place slowly in the solutions under ordinary conditions, can be produced, like optical stability, almost instantaneously by the addition of traces of an alkali. A full account is given of the apparatus employed, of the method of experiment, and the nature of the corrections to be applied. In the cases of dextrose and milk-sugar, there is a liberation of heat accompanying the change of rotatory power; with lævulose there is a very decided absorption; and with maltose no thermal disturbance is recognisable. The following are the values obtained :—

	Per grm. of sugar.	Per grm.-molecule.
Dextrose ..	+0.588 cal.	+106 cal.
Lævulose ..	-4.64 cal.	-835 cal.
Milk-sugar ..	+0.19 cal.	+ 34 cal.
Maltose ..	0	0

The authors discuss the various explanations which have been given from time to time to account for multi-rotation, and consider that their experiments favour the view that it is conditioned by chemical rather than physical causes, and that Fischer is probably correct in his suggestion that dextrose, for instance, in passing from the optically unstable to the optically stable modification in solution, passes from the aldehyd, $C_6H_{12}O_6$, to the heptahydric alcohol, $C_6H_{14}O_7$. They believe, however, that the analogy which Fischer suggested, of the change of a lactone into its acid, is less close than that afforded by the gradual change of acetic aldehyd, in contact with water, into ethylidene glycol where the CHO group becomes $CH(OH)_2$.

*68. "On the Thermo-chemistry of Carbohydrate Hydrolysis. (I.) *The Hydrolysis of Starch by Vegetable and Animal Diastase*. (II.) *The Hydrolysis of Cane-sugar by Invertase*." By HORACE T. BROWN, F.R.S., and SPENCER PICKERING, F.R.S.

The attempts made to determine the thermal effects of hydrolysis have hitherto been confined to indirect methods, based on the heats of combustion of the hydrolysable substance and its products. Such methods, it is shown, cannot give results of any real value, as the thermal changes to be measured are considerably within the experimental errors of the combustion values.

The paper describes the results obtained by direct measurement of the heats of hydrolysis of starch and of cane-sugar. Lintner's soluble starch was for the most part used, as there are certain mechanical difficulties in employing starch paste in the calorimeter, owing to its viscosity. The hydrolytic agents used for starch were (1) malt-diastase, (2) pancreatic diastase, (3) Taka-diastase, and (4) saliva. With malt-diastase, the heat of hydrolysis was found to be +2.60 calories per grm. of amylin converted into maltose. The amount of heat is proportional to the water fixed, and is independent of the molecular complexity of the amylin attacked. The breaking down of the starch molecule prior to hydrolysis does not appear to be attended with any thermal disturbance.

With pancreatic diastase, the heat liberated per grm. of amylin hydrolysed amounts to +1.8 cal., a value sensibly less than that deduced from the action of the malt diastase. With Taka-diastase, the heat disturbance is still less than with the other two agents. The possible causes of these differences are discussed.

Cane-sugar was hydrolysed with invertase, and was found to give a thermal effect of +11.21 cal. per grm. of cane-sugar inverted when the products were in the optically stable β -form, and 13.34 cal. per grm. at the moment of liberation of the products, i.e., when they are in their "birotatory" or optically unstable form. It is the larger number which correctly represents the heat of hydrolysis of cane-sugar.

DISCUSSION.

Mr. PICKERING made a statement as to some additional work which had been done in connection with the subject since the paper had been sent in to the Society. The nature of the change produced by water on the sugars has been suggested to be the conversion of the aldehyd groups present into aldehydrol groups, an action which there is every reason to believe occurs in the case of acetic aldehyd itself, and experiments were, therefore, made to ascertain whether the action in the case of aldehyd exhibits the same peculiarity as in the case of the sugars, of being greatly accelerated by the addition of alkali. This was found to be the case, and the analogy of the two actions is, therefore, greatly strengthened. In both cases, also, the ammonia combines directly with the sub-

stance—the sugar or the aldehyd—with evolution of heat, but the results with aldehyd show that the formation of these compounds is not the cause of the hydrating effect of the alkali, for, although the alkali renders the hydration instantaneous, the formation of the aldehyd ammonia proceeds gradually, and with the quantities used is complete only after seven or eight minutes, the action evidently being independent of, and posterior to, the hydration. The accelerative action of the alkali on the hydration is probably due to its increasing the number of free molecules of water present in the liquid, by forming continually dissociating compounds such as NH_4OH , $NaOH \cdot H_2O$, &c., and a free molecule of water would be a far more active hydrating agent than the average water aggregate constituting the bulk of liquid.

Mr. A. R. LING said he was under the impression that Béchamp, and subsequently Tollens, were the first to point out that the multirotation of carbohydrates was correlated with thermic phenomena, but neither had made the exact measurements now presented. He wished to know if the authors had observed any change in the density of the solutions before and after the transition from the abnormal to the normal rotatory power.

Dr. KIPPING said that although it seems to be generally understood that the phenomenon of birotation is not the result of a purely physical change, the assumption that it is due to the mere hydration of the aldehyd group might be objected to as involving the apparent contradiction that a very considerable change in specific rotation is brought about, not by increasing or diminishing the number of asymmetric carbon atoms in the molecule, but by merely altering to a comparatively slight extent the asymmetry of the groups already present. Important data in support of the chemical or hydration explanation of birotation were afforded, however, by some experiments made at the suggestion of Emil Fischer, as it had been found (Jacobi, *Ann.*, 1892, cclxxii., 170) that the rapidity of the formation of a hydrazone from a sugar which showed birotation varied with the time which had elapsed since the sugar had been dissolved.

Mr. HORACE BROWN, in reply, said that he believed Mr. Ling was mistaken in his statement that Béchamp or Tollens had done anything to correlate multirotation with thermic phenomena. So far as he knew, the only previous work on this subject was that of Berthelot, who had indirectly determined, for the solid state, the heats of transformation of α - and γ -dextrose into the β form.

Up to the present time, the authors had been unable to discover any change of density in solutions of multi-rotatory sugars.

Whilst fully admitting the force of Dr. Kipping's objections, it must be remembered that, so far as our knowledge goes at present, we are not justified in denying that comparatively small changes in the asymmetry of groups may be attended with a considerable change in rotatory power. The experiments of Jacobi are fully described in the paper, and are regarded by the authors as strongly confirmatory of their views.

(To be continued).

PHYSICAL SOCIETY.

Ordinary Meeting, June 11th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

A MATHEMATICAL paper was read by Mr. C. S. WHITEHEAD, on "The Effect of Sea-Water on Induction Telegraphy."

If a secondary circuit containing a telephone is rightly placed with respect to the field of a primary circuit traversed by an alternating current, signals may be transmitted over considerable distances. The author investigates the effect of filling the intervening space with sea-water; and, generally, the effect of a spherical conducting shell

on the induction, at a point in a dielectric, due to an alternating current in a circular circuit, when the axis of the conductor passes through the centre of the shell. In the mathematical treatment two cases are considered:— (1) To find the normal magnetic induction at any point in the dielectric outside the shell when a circular circuit carrying an alternating current is placed in the dielectric inside a spherical conducting shell. (2) To find the normal magnetic induction at any point on the remote side of an infinite conducting plate, due to a circular circuit parallel to the plate. In both cases the following result is arrived at:—

$$\frac{v_0}{u_0} = e^{-q\eta},$$

where v_0 is the maximum value of the normal magnetic induction at any point outside; u_0 the maximum normal magnetic induction due to the current in the primary, supposing the conducting shell or plate absent, at the same point; η the thickness of the shell or plate; and—

$$q = \left(\frac{2\pi\mu p}{\sigma} \right)^{\frac{1}{2}}$$

where μ is the permeability of the conducting shell or plate; σ its specific resistance, $p = 2\pi$ times the frequency. If the frequency is 300, $p = 1885$. For sea-water, σ is taken as 2×10^{10} C.G.S. units, and $\mu = 1$. The sea-depth at the North Sand Head corresponds to $\eta = 2000$ c.m. Hence, in this case,—

$$\frac{v_0}{u_0} = 0.21,$$

or 79 per cent is lost. Similarly, when $\eta = 1000$, the loss is 54 per cent. The method employed in the investigation is that suggested by Lamb and Niven; the author adds an expression for Ω , the solid angle subtended by the circuit at any point, in terms of Bessel's functions.

Mr. EVERSHED referred to some experiments of his own, from which he concluded that the author's formula gave too low an estimate of the attenuation; the discrepancy indicated that some term had been neglected.

Mr. YULE doubted whether the equations given by the author were quite applicable to sea-water. There was need, apparently, of a term involving the polarisation of the medium.

Mr. HEAVISIDE communicated a criticism of the paper. It was not necessary to investigate the problem for any particular form of circuit from which the waves proceed. The attenuating factor for plane waves, due to Maxwell, was sufficient. Taking the best-known value for the conductivity of sea-water, there was no reason why the conductivity should interfere with signalling. A considerably greater conductivity must be proved for sea-water before it could be accepted that the failure of experiments on telegraphic communication with light-ships from the sea-bottom was due to that factor. It was unlikely theoretically, and Mr. Stevenson had contradicted it from a practical stand-point. For some reason, the account of the light-ship experiments had not been published, so that there was no means of finding the real cause of failure.

Mr. T. H. BLAKESLEY read a paper on "*A New Definition of Focal Length, and an Instrument for Determining it.*"

The author asserts the principle that the focal length of a lens-combination is an abstract quantity, not necessarily the distance between two particular points. It is a quantity best defined in terms of some function of the two distances of object and image from their appropriate focal centres. Such a function is the magnification factor, m , the linear ratio of image to object, positive if the image is erect with regard to the object. Consider a particular pair of conjugate foci on the axis of a lens-system. Let one of these foci be at distance v from some fixed point

on the axis, measured positively, in the direction of the rays. Then—

$$\frac{dv}{dm}$$

is constant, and is the focal length, f . If v_0 is the value of v when $m = 0$, $v - v_0 = f \cdot m$.

Let u be the position of the other focus, and u_0 its value when $m = \infty$. Then $u - u_0 = f \cdot m^{-1}$; and—

$$\frac{v - v_0}{u - u_0} = m^2.$$

The last expression, m^2 , may be called the "areal magnification"—it is important in determining photographic exposure. The author describes an optical bank, which enables—

$$\frac{dv}{dm}$$

to be measured by a very simple operation; it gives also a record, on a paper strip, of the magnification factor corresponding to various relative positions of object and image.

Dr. S. P. THOMPSON said the paper was the most important contribution to geometrical optics that had appeared for many years. The introduction of the magnification function was a most useful device, leading to exceedingly simple results. The important thing to measure was not so much the focal length as the reciprocal of that quantity.

Dr. CHREE said the photographic method at present used at Kew for determinations of focal length gave greater security than any more direct method. The colour of the light had to be taken into account.

Mr. BLAKESLEY, in replying, called attention to the use of his strip diagrams of magnification, for enlarging purposes in photography. When the magnification along some definite line was known, the focussing-cloth might almost be dispensed with.

Dr. J. A. FLEMING read a paper on "*A Method of Determining Magnetic Hysteresis Loss in Straight Iron Strips.*"

The author's process is based upon the use of the bifilar reflecting Watt-meter. The samples of iron, large or small, in the form of straight strips are inserted in a long solenoid. The solenoid is traversed by an alternating current, and the square-roots of the mean-square values of the current are determined by a Kelvin balance. A flat bobbin of fine wire may be slid along the strip; an electrostatic voltmeter connected to the ends of this exploring coil gives the square-roots of the mean-square values of the electromotive force in that coil. From these measurements, and the known dimensions of the solenoid and coil, the induction-density, B_1 , can be found at any point of the length of the strip. From these results a curve is drawn, co-ordinating the values of B to corresponding distances along the half-length of the strip. Assuming the hysteresis loss per cycle, per c.c. of iron, to vary as the 1.6th power of the maximum induction density, and then raising all the B ordinates to the 1.6th power, and plotting a new curve over the first, another curve is obtained which represents the variation of hysteresis loss per c.c. of iron from point to point along the half-length of strip. Now at some point along the half-length of strip there must be a section where the induction density is B_1 , such that the true mean hysteresis loss for the whole bar is proportional to $B_1^{1.6}$. Let this value of the induction density be called the "effective value" and the corresponding point in the strip the "effective point." Let $M \cdot B_1^{1.6}$ stand for the mean ordinate of the curve representing the varying values of $B^{1.6}$ all along the half-length. Then, evidently,—

$$B_1 = \sqrt[1.6]{M \cdot B^{1.6}}.$$

The following curious experimental result is found:—Whatever may be the length or section of the iron strip, the point at which the actual induction density has a value equal to the "effective" value always comes at the same proportional distance from the centre of the strip. This distance is very exactly equal to 0.56 of the half-length, as measured from the middle, or 0.22 of the whole length from one end. If, therefore, the secondary coil is placed at that spot, and the secondary voltage then observed is used to calculate the induction density, the value so obtained corresponds to the true mean value of the varying hysteresis loss per c.c. all along the strip.

Mr. CARTER asked whether roots other than the 1.6th gave a similar constant value of the induction density.

Dr. FLEMING said it seemed to be the result of accident that the 1.6th root gave a constant value for iron.

The PRESIDENT proposed a vote of thanks to the authors, and the meeting adjourned until June 25th.

THE ROYAL SOCIETY.

THE Conversazione held on Wednesday, the 16th inst., at the Royal Society's Rooms, Burlington House, was in every respect a most brilliant and interesting gathering.

Apart from the purely social side of the entertainment, there were a number of exhibits which could not fail to attract attention. On entering, the eye was immediately caught by Prof. RAMSAY's Argon and Helium Tubes, arranged to form the words "Vivat Victoria Regina."

In the Office upstairs, some of Lord KELVIN's Experiments on the Quasi-perpetual Motion Action, of Uranium-Zinc Contact were shown.

One of the most striking exhibits was that of Dr. ALEX. MUIRHEAD, who had an actual working instrument receiving, by means of a Kelvin syphon recorder, the signals transmitted from a distant room through the surging crowd, and being recorded on the tape in dots and dashes without the help of any intervening wires.

Prof. ROBERTS-AUSTEN showed some Photographs and Microscopic Slides of Diamonds separated from carburised iron, made, with certain modifications, according to the method described by M. Moissan.

The Shadow Photographs illustrating the absorption of X rays by certain elements and their compounds, exhibited by Dr. J. H. GLADSTONE and Mr. WALTER HIBBERT, were of peculiar interest. The "photographs" were obtained by subjecting various tubes, containing equal parts of certain elements, but in different compounds, to the action of the X rays, and it was noted in the case of carbon that the absorption was the same whether the tubes contained charcoal, anthracene, or naphthalene; the different forms of combination of the carbon having no influence whatever on the result.

A beautiful example of the work of our almost prehistoric forerunners was shown by Sir JOHN EVANS. It consisted of a collection of flint knives and lance-heads from Egypt, dating back to 4000 B.C. These exquisitely fluted blades have been polished all over by grinding before the flakes were removed; the edges were subsequently re-touched and serrated with minute teeth at intervals of about 35 to the inch.

Medals, struck in gold, silver, and bronze, to commemorate the 60th year of the reign of Her Majesty the Queen, were exhibited by Mr. HORACE SEYMOUR, Deputy Master of the Mint. These attracted a good deal of attention.

At 9.45 and 11 o'clock Demonstrations of Signalling through Space without Wires were given by Mr. W. H. PREECE, C.B.; and at 10.30 Photographs illustrating the arrangements of the 1896 Eclipse Expedition at Kiō and Novaya Zemlya, were exhibited by Mr. J. NORMAN LOCKYER, C.B., in the meeting room on the ground floor.

NOTICES OF BOOKS.

Electro-chemical Problems for Practice and for Self-instruction. Collected by Dr. FELIX OETTEL. With 20 Woodcuts in the Text. Halle-on-Saale: W. Knapp. 1897. 8vo., pp. 53.

AN unusual degree of attention seems to be at present directed to electro-chemistry.

The work before us opens with generalities on the necessary arrangements. As a source of current the preference is given to accumulators which may either be charged by means of a small dynamo or from a central station. For electro-metallurgical investigations a tension of 4 volts (= 2 accumulators) is generally sufficient. For regulation, a crank regulator with 15 to 20 contacts is considered convenient.

As measuring instruments the author recommends those of Meyer, form E, which are accurately gauged notwithstanding their cheapness (25 marks), though the Weston instruments (made apparently by a Berlin firm) have advantage of rapid adjustment, but are decidedly more expensive than the apparatus above mentioned.

The electro-chemist is strongly advised to test the accuracy of his measuring instruments.

All the instruments above mentioned are unaffected by currents in their neighbourhood, even by dynamos in action. But the instruments which work with magnetic needles are much less widely useful. They must be placed at least at the distance of some metres from other circuits.

A galvanometer on the system of Deprez d'Arsonval is not affected by neighbouring currents, and may be set up anywhere.

The most important voltmeters are the copper voltmeter and the detonating-gas instrument. The solution for the former is made up of 150 grms. blue vitriol, 50 grms. sulphuric acid, 50 grms. alcohol, and 1000 grms. water. The liquid must be agitated during use.

The detonating-gas voltmeter, which is permanently inserted in the circuit, is the most convenient instrument for gauging and checking ammeters. This apparatus, as usually constructed, consists of a stout glass cylinder with two concentric sheets of nickel, the internal one serving always as anode.

In a section on testing and gauging measuring appliances, there is first a comparison between the silver and the copper voltmeters, the former of which is universally regarded as the most accurate. Then follows the checking of the ammeter respectively with the detonating-gas voltmeter and the copper voltmeter, the comparison of the ammeter with the tension galvanometer, the duplication of the measuring scope of an ammeter by a by-connection.

The gauging of a galvanometer respectively as ammeter and as voltmeter forms the subject of two short but clearly written sections.

We next come to the dependence of the tension of the bath upon the following factors: density of current concentration and temperature of the solution, and mutual distances of the electrodes. There is a special discussion on the influence of density of current and concentration upon the course of electro-chemical reaction.

Oxidation is most favourable in a concentrated solution, and with a low density of anodic current.

Reduction is promoted in a concentrated solution, but with a low cathodic current.

Oxidation is slightest in a dilute solution, and with a high density of anodic current.

The slightest reduction ensues in dilute solutions, and with a high density of current at the kathode.

In accounts on electro-chemical work a statement of the density of the current at each pole is therefore necessary. We quote this statement literally, as a short time ago it was the subject of a discussion.

Next follow the oxidation of oxalic acid; the electrolysis of a mixed ferrous and ferric solution; the formation of hypochlorites and chlorates; and the electrolysis of hydrochloric acid without a diaphragm.

The last sections of the work treat of the precipitation of metals with soluble and insoluble anodes; the introduction of auxiliary reactions of experiments with pyro-liquid electrolytes; the separation of copper with bipolar electrodes; the electrolysis of sodium acetate, and other organic electrolyses.

Dr. Oettel has favoured the chemical world with a very useful, compact, and well-illustrated work.

CORRESPONDENCE.

ESTIMATION OF CARBON IN FERRO-CHROME.

To the Editor of the Chemical News.

SIR,—I have neither time nor inclination to follow Mr. R. L. Leffler into the details of his letter. Mr. Leffler stated that my method gave only half the carbon present in ferrochrome: this statement was inaccurate. That Mr. Leffler has been putting up men of straw to somewhat ostentatiously knock them down again will be seen from Mr. Saniter's letter. As till quite recently Mr. Leffler was a student of mine, it would seem that the dictates of common courtesy would have led him to ask personally for an explanation of his low results before making the rash assertion in the article. No amount of ingenuity of argument on Mr. Leffler's part will alter the fact that other students have obtained from 8 to 9 per cent of carbon from ferro-chrome by the method which in his hands failed.—I am, &c.,

J. O. ARNOLD.

The Technical Dept., University College,
Sheffield, June 12, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 19, May 10, 1897.

The President announced two severe losses which the Academy has experienced. The illustrious mineralogist M. des Cloiseaux, one of the oldest members of the Academy, is dead. He was born in 1817, and was elected a Member of the Academy in 1869. His principal researches were devoted to crystallography and to the optical properties of minerals. The Duc d'Aumale was indirectly one of the victims to the terrible catastrophe of the Rue Jean Goujon, in which his niece, the Duchess d'Alençon, perished. He bequeathed to the Institute his estate of Chantilly, formerly the residence of the Princes de Condé, which he had fitted up as a library and museum of art.

Explanation of some Experiments of G. le Bon's. H. Becquerel.—Already inserted.

The Solutions of Acetylene, and on their Explosive Properties.—MM. Berthelot and Vieille.

Remarks on the Explosive Decomposition of Solutions of Acetylene.—MM. Berthelot and Vieille.

Some Conditions of the Decomposition of Pure Acetylene.—MM. Berthelot and Vieille.

Solubility of Liquids.—A. Aignon.—One litre of the ether examined can dissolve 33 c.c. of water, whilst one litre of water can dissolve 132 c.c. of the same ether.

On Multiple Resonance.—L. Deconté.—This paper requires the two accompanying figures.

Diurnal Variation in the Direction of the Wind.—Alfred Angot.—This memoir also necessitates the accompanying illustration.

Basic Salts of Cadmium.—M. Tassilly.—The action of metallic oxides upon the corresponding haloid salts has yielded two novel cadmium compounds, an oxybromide and an oxyiodide. These bodies have been obtained by heating to 200°, in a sealed tube, a concentrated solution of bromide or iodide along with cadmium oxide. The yields are exceedingly small. The bodies obtained are distinctly crystalline, and act upon polarised light. The oxyiodide gave on analysis the formula $\text{CdI}_2\text{CdO}_3\cdot\text{H}_2\text{O}$. It is little acted on by water. At 120° it does not vary in weight either in a current of nitrogen or of air deprived of carbonic acid and watery vapour. In parallel light the crystals present an extinction parallel to the longitudinal axis. In converging light we observe a lemniscate. The crystal is bi-refrangent at axes widely remote from each other. The oxybromide appears in very small crystals, answering to the formula $\text{CdBr}_2\text{CdO}_3\cdot\text{H}_2\text{O}$, and acting upon polarised light. In conclusion it is well to observe that the basic salts of cadmium are always formed of equal molecules, contrary to what takes place with the salts of zinc, which are capable of fixing a variable number of mols. of oxide to form salts which do not answer to a general type.

Researches on Strontium Sulphide, and a Method of Rendering it Highly Phosphorescent.—José R. Movelo.—After many trials I had the idea of modifying the procedure previously adopted, and I had recourse to a method by which I obtained a strontium sulphide possessing a magnificent phosphorescence of a greenish blue and so intense that, after insolation for less than a second, it was perceptible by its shade without the necessity of placing the substance in the dark. I took 285 grms. of impure commercial strontium carbonate, 62 grms. flower of sulphur, 4 grms. crystalline sodium carbonate, 2.5 grms. sodium chloride, and 0.4 gm. bismuth subnitrate. The mixture, finely powdered, was placed in an earthen crucible, pressed down and covered with a layer of tinder in coarse powder: this stratum does not exceed 2 c.m. in depth. The crucible, set in a furnace, is heated to bright redness by a coke fire for five hours, and is then allowed to cool slowly for ten or twelve hours. After this we extract from the crucible an agglomerate, nearly white, granular, and friable, possessing a phosphorescent power which the diffused light of the laboratory is sufficient to excite in the shade and behind the windows of the cupboard in which the bottle was inclosed. Like M. Verneuil I have observed that most of the strontium sulphides which I have prepared lose their phosphorescent power if powdered, but these pulverised sulphides, if mixed with tinder and heated to bright redness for five hours, resume their phosphorescent power.

Thermic Study of Mono- and Di-sodic Acetylenes.—Camille Matignon.—A thermo-chemical paper, suitable neither for abstraction nor for insertion in full.

Contribution to the Study of the Preparation of Common Ether.

Action of Chlorine Hydrate upon Phenylhydrazinodiphenylglyoxazol and its Derivatives.—H. Causse.—The compounds here described are trichlorethylidenediphenylhydrazin, chlorodiphenylglyoxazol, hydroxydiphenylglyoxazol, oxydiphenylglyoxazol antimonite, and the barium derivative.

Intervention of Manganese in the Oxidations induced by Laccase.—G. Bertrand.—Already inserted.

No. 20, May 17, 1897.

On the Cathodic Rays and on certain Phenomena in Vacuum Tubes.—The tube used by the authors was pear-shaped; the electrodes terminating in two aluminium discs, one of which was placed at the narrow extremity of the tube, whilst the other was placed in the expanded

part, the surfaces of the two discs being respectively perpendicular. At the anti-cathode there is formed a luminous ring and a central spot. If the disc in the narrow part serves as a cathode, the following phenomena have been observed:—If we touch with the finger the tube near the cathode disc we observe an attraction of the cathodic rays. The glass under the finger is rendered luminous and all the cathodic bundle is deflected towards the hand. We observe at the same time that under the anticathodic ring the phosphorescence becomes stronger towards the finger and the central spot undergoes a transformation from circular towards elliptical, as if subjected to pressure. There is in front of the anode a bluish light which, if examined with the spectroscopic, gives the line-spectrum of nitrogen. If we touch the tube with the finger at a point of the expanded portion, the anodic light is energetically repelled.

Transparence of Ebonite.—M. Perigot.—The phenomena ascribed to "black light" are explained by the well-known fact of photographic inversion.

Lithium Borate.—The composition of this salt is $\text{Bo}_2\text{O}_3, \text{LiO}, 16\text{H}_2\text{O}$. Its specific gravity at 14.7° is 1.397. Its crystallisation in the rhombohedral system is distinctly uniaxial. Its heat of hydration is $+43.4$ cal. Its solution heat is -28.4 cal. Crystalline lithium borate effloresces slowly if left in contact with the air, and at the same time absorbs small quantities of carbonic acid.

Alloys of the Silver and Copper Group.—F. Osmond.—M. Charpy, on examining microscopically different groups of alloys, has established that the structure of the eutectics recalls generally that of the perlitic of steels, and the comparison is justified from all points of view. Micrography confirms absolutely the indications of the curve of fusibility, and leaves no doubt concerning the non-existence of Ag_3Cu_2 as a definite compound.

Researches on the Colouration of Glasses by the Direct Penetration of Metals or Metallic Salts.—Léon Léal.—The author applies upon glass a salt of silver and raises the temperature to $500-550^\circ$. The glass, when cooled and freed by washing from excess of salt, has a yellow colour. The shade obtained may range from straw-colour to an orange-red. Glasses thus coloured present phenomena of dichroism always yellow by transmitted light, but having by reflected light fluorescent tints, from yellowish green to violet-blue.

Action of Water upon Phosphoryl Chloride.—A. Besson.—The limited action of water upon POCl_3 yields the series of products $\text{P}_2\text{O}_3\text{Cl}_4, \text{PO}_2\text{Cl}$ and PO_4H_3 , in virtue of the reactions $2\text{POCl}_3 + \text{H}_2\text{O} = 2\text{HCl} + \text{P}_2\text{O}_3\text{Cl}_4$.

Certain New Aromatic Synthetic Ureas.—P. Cazeneuve and M. Mar.—A description of dipseudocumyl urea, dixyl urea, dipara-aminyl urea. These results lead to the conclusion that all the primary bases yielding with guaiacal carbonate corresponding symmetrical ureas are very easy to obtain.

Amidised Amidines.—Charles Lauth.—The hydrochlorates of these two amidines are easily diazotised. The diazoics obtained yield, on conjugation with the phenols and the amines, azo-pigments which dye cotton directly, in shades ranging from yellow to red and black. They resist well the action of chemical reagents, but they are not very fast.

Rôle of the Tannins in Plants and especially in Fruits.—C. Gerber.—In fruits containing tannins—such as the kakis, these tannins disappear by complete oxidation without giving rise to carbohydrates.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 14th inst., Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, presiding. The following were elected Members:—Dr. Tempest Anderson, M.D.; Mr. Samuel Pope, Q.C.; and Major Clifford Probyn.

MEETINGS FOR THE WEEK.

FRIDAY, 25th.—Physical, 5. "A New Theory of the Earth's Magnetism," by Mr. Sutherland. "Experiments in Critical Phenomena," by Dr. Kuenen. "Attenuation of Electric Waves in Wires," by Dr. Barton. "The Steady Motion of an Electrified Ellipsoid," by G. F. C. Searle.

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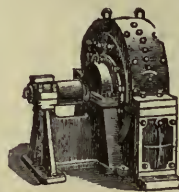
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THE CHEMICAL NEWS.

VOL. LXXV., No. 1961.

DIAMONDS.*

By WILLIAM CROOKES, F.R.S., M.R.I.

It seems but the other day I saw London in a blaze of illumination to celebrate Her Majesty's happy accession to the throne. As in a few days the whole Empire will be celebrating the Diamond Jubilee of our Queen, who will then have reigned over her multitudinous subjects for sixty years, what more suitable topic can I bring before you than that of Diamonds! One often hears the question asked: "Why *Diamond Jubilee*?" I suppose it is a symbol intended to give a faint notion of the pure brilliancy and durability of the Queen's reign; and in thus associating Her Majesty with the precious Diamond, to convey an idea of those noble qualities public and private which have earned for her the love, fealty and reverence of her subjects.

From the earliest times the diamond has occupied men's minds. It has been a perennial puzzle—one of the riddles of creation. The philosopher Steffans is accredited with the dictum that "Diamond is quartz which has arrived at self-consciousness;" and an eminent geologist has parodied this metaphysical definition, saying—"Quartz is diamond which has become insane!"

Professor Maskelyne, in a lecture "On Diamonds," thirty-seven years ago,† in this very theatre, said:—"The diamond is a substance which transcends all others in certain properties to which it is indebted for its usefulness in the arts and its beauty as an ornament. Thus, on the one hand it is the hardest substance found in nature or fashioned by art. Its reflecting power and refractive energy, on the other hand, exceed those of all other colourless bodies, while it yields to none in the perfection of its pellucidity"—but he was constrained to add "The formation of the diamond is an unsolved problem."

Recently the subject has attracted many men of science. The development of electricity, with the introduction of the electric furnace, has facilitated research, and I think I am justified in saying that if the diamond problem is not actually solved, it is certainly no longer insoluble.

In the early part of last year, accompanied by my wife, I visited some of our Colonies in South Africa, and spent a considerable time in the neighbourhood of the famous Diamond Mines of Kimberley, where I had an exceptionally good opportunity of studying the peculiar geological formation, and of noting interesting facts connected with the occurrence of the precious stone which forms the subject of this evening's lecture.

Although the experiments I wish to bring before you are chiefly connected with the physical and chemical properties of diamonds, and of the light that recent researches throw upon their probable formation, it will possibly act as a kind of compensation for the dryness of some of the theoretical points if with the help of a few photographs taken on the spot, I bring before your very eyes the general character of the famous mines and their surroundings.

The most famous diamond mines are Kimberley, De Beers, Dutoitspan, Bulfontein, and Wesselson. They are situated in latitude $28^{\circ}43'$ South, and longitude $24^{\circ}46'$ East. Kimberley town is 4042 feet above sea-level. Other mines in the district, as yet unimportant, are worked for diamonds. Kimberley is practically in the

centre of the present diamond-producing area. Besides these mines, there are in the Orange Free State, about 60 miles from the Kimberley diamond region, two others of some importance known as Jagersfontein and Coffeefontein.

River Washings.

Before describing the present mode of diamond extraction followed in the leading mines, I will commence with the so-called "River Washings," where, in their original simplicity, can be seen the methods of work and the simple machinery long since discarded in the large centres. These drifts or "river-washings" present an interesting phase of Diamond industry. The work is carried out in the crude fashion of early diamond discovery, every man working on his own little claim, assisted by a few natives, and employing primitive machinery. The chief centre of the river washings is at Klipdam No. 2, about 30 miles to the north-west of Kimberley. The road to Klipdam No. 2 involves a journey of about a dozen miles in one of the old African coaches now becoming obsolete through the spread of railways. Road there is none—only a track across the veldt made by countless teams of oxen and mules.

Diamonds from the "river washings" are of all kinds, as if every mine in the neighbourhood contributed. The samples are much rolled and etched, and contain a fair proportion of stones of very good quality, as if only the better and larger stones had survived the ordeal of knocking about.

Diamonds from the drift fetch about 40 per cent more than those from Kimberley: taking the yield of the Kimberley and De Beers mines as worth, all round, large and small, 26s. 6d. a carat, the drift diamonds are worth 40s.

Kimberley.

The town of Kimberley is a remarkable instance of rapid growth. It has an excellent club and one of the best public libraries in South Africa. Parts of the town, affectionately called "the camp" by the older inhabitants, are still in the galvanised iron or "tin shanty" stage, and the general appearance is unlovely and depressing. Reunart reckons that over a million trees have been felled to supply timber for the mines, and the whole country within a radius of 100 miles has been denuded of wood with most injurious effects to the climate. The extreme dryness of the air, and the absence of trees to break the force of the wind and temper the heat of the sun, probably account for the dust storms so frequent in summer. The temperature in the day frequently rises to 100° in the shade, but in so dry a climate this is not unpleasant, and I felt less oppressed than I did in London the previous September. Moreover, in Kimberley, owing to the high altitude, the nights are always cool.

The Pipes.

The five noted diamond mines are all contained in a circle $3\frac{1}{2}$ miles in diameter. The mines are irregularly-shaped round or oval pipes, extending vertically downwards to an unknown depth, retaining about the same diameter throughout. They are said to be volcanic necks, filled from below with a heterogeneous mixture of fragments of the surrounding rocks, and of older rocks such as granite mingled and cemented with a bluish-coloured hard clayey mass, in which famous blue the diamonds are hidden.

The breccia filling the pipes, usually called "blue ground," is a collection of fragments of shale, eruptive rocks, boulders, and crystals of many kinds of minerals.

The Kimberley mine for the first 70 or 80 feet is filled with what is called "yellow ground," and below that with "blue ground." This superposed yellow on blue is common to all the mines. The blue is the unaltered ground, and owes its colour chiefly to the presence of lower oxides of iron. When atmospheric influences have access to the iron it becomes peroxidised, and the ground

* A Lecture delivered at the Royal Institution, Friday, June 12th, 1897.
† CHEMICAL NEWS, vol. i., p. 208.

assumes a yellow colour. The thickness of yellow earth in the mines is therefore a measure of the depth of penetration of air and moisture. The colour does not affect the yield of diamonds.

The diamantiferous clay or blue ground shows no signs of passing through great heat, as the fragments in the breccia are not fused at the edges. The eruptive force was probably steam or water-gas, acting under great pressure but at no high temperature. According to Mr. Dunn, in the Kimberley mine, at the depth of 120 feet, several small fresh-water shells were discovered in what appeared to be undisturbed material.

Let me cite a description of a visit to Kimberley in 1872, by Mr. Paterson, taken from a paper read to the Geologists' Association, which gives a graphic picture of the early days of the Kimberley mine:—

"The New Rush diggings (as the Kimberley mine was first called) are all going forward in an oval space enclosed around by the trap dyke, of which the larger diameter is about 1000 feet, while the shorter is not more than 700 feet in length. Here all the claims of 31 feet square each are marked out with roadways about 12 feet in width, occurring every 60 feet. Upon these roadways, beside a short pole fixed into the roadway, sits the owner of the claim with watchful eye upon the Kafir diggers below, who fill, and hoist by means of a pulley fixed to the pole above, bucketful after bucketful of the picked marl stuff in which the diamonds occur."

Soon came the difficulty how to continue working the host of separate claims without infringements. A system of rope haulage was then adopted. This mode of haulage continued in vogue during the whole of 1873, and if the appearance of the mine was less picturesque than when roadways existed, it was by moonlight, particularly, a weird and beautiful sight.

But the mine was now threatened in two other quarters. The removal of the blue ground undermined the support from the walls of the pipe, and frequent falls of reef occurred, not only burying valuable claims, but endangering the lives of workers below. Moreover, as the workings deepened, water made its appearance, necessitating pumping.

It soon became evident that open workings were doomed, and by degrees the present system of underground working was devised.

During this time of perplexity, individual miners who might have managed one or two claims near the surface could not continue work in the face of harassing difficulties and heavy expenses. Thus the claims gradually changed hands until the mine became the property first of a comparatively small number of capitalists, then of a smaller number of limited liability companies, until the whole of the mines have practically become the property of the "De Beers Consolidated Mines, Limited."

The areas of the mines are:—

Kimberley	33 acres.
De Beers	22 "
Dutoitspan	45 "
Bulfontein	36 "

The contents of the several pipes are not absolutely identical. The diamonds from each pipe differ in character, showing that the upflow was not simultaneous from one large reservoir below, but was the result of several independent eruptions. Even in the same mine there are visible traces of more than one eruption.

The blue ground varies in its yield of diamonds in different mines, but is pretty constant in the same mine. In 1890, the yield per load of blue ground was—

From the Kimberley mine	from 1'25 to 1'5 carats.
„ De Beers mine	„ 1'20 „ 1'33 „
„ Dutoitspan mine	„ 0'17 „ 0'5 carat.
„ Bulfontein mine	„ 0'5 „ 0'33 „

Underground Workings.

In the face of constant developments I can only describe the system in use at the time of my visit. Shafts are sunk in the solid rock at a sufficient distance from the pipe to be safe against reef movements in the open mine. Tunnels are driven from this shaft at different levels, about 120 feet apart, to cross the mine from west to east. These tunnels are connected by two others running north and south, one near the west side of the mine and one midway between it and the east margin of the mine. From the east and west tunnels offsets are driven to the surrounding rock. When near the rock, the offsets widen into galleries, these in turn being stoped on the sides until they meet, and upwards until they break through the blue ground. The fallen reef with which the upper part of the mine is filled sinks and partially fills the open space. The workmen then stand on the fallen reef and drill the blue ground overhead, and as the roof is blasted back the *débris* follows. When stoping between two tunnels the blue is stoped up to the *débris* about midway between the two tunnels. The upper levels are worked back in advance of the lower levels, and the works assume the shape of irregular terraces. The main levels are from 90 to 120 feet apart, with intermediate levels every 30 feet. Hoisting is done from only one level at a time through the same shaft. By this ingenious method of mining every portion of blue ground is excavated and raised to the surface, the rubbish on the top gradually sinking and taking its place.

The scene below ground in the labyrinth of galleries is bewildering in its complexity, and very unlike the popular notion of a diamond mine. All below is dirt, mud, grime; half naked men, black as ebony, muscular as athletes, dripping with perspiration, are seen in every direction, hammering, picking, shovelling, wheeling the trucks to and fro, keeping up a weird chant which rises in force and rhythm when a titanic task calls for excessive muscular strain. The whole scene is more suggestive of a coal mine than a diamond mine; and all this mighty organisation, this strenuous expenditure of energy, this costly machinery, this ceaseless toil of skilled and black labour, goes on day and night, just to win a few stones wherewith to deck my lady's finger.

(To be continued).

ON THE ACTION EXERTED BY CERTAIN METALS AND OTHER SUBSTANCES ON A PHOTOGRAPHIC PLATE.

By W. J. RUSSELL, Ph.D., F.R.S.,
Lecturer on Chemistry at St. Bartholomew's Hospital.

HAVING some years ago prepared for the purpose of spectroscopic examination several uranium compounds, it was of interest to make further use of them by repeating some of the very important experiments which Becquerel has made with these compounds. He has shown that if the metal or some of its salts be placed on a photographic plate in perfect darkness, and allowed to remain there for some days, the plate becomes acted on, the action being rendered evident by the ordinary photographic process of development. This action is readily produced, and belongs apparently to all the salts of this metal, and, as Becquerel has shown, to uranous as well as uranic salts. It is very remarkable that this power belongs also to the salts when in solution, and, as the action passes through glass, solutions of the double chloride or of the nitrate contained in a thin glass bottle, when placed on a photographic plate, act readily upon it. While speaking of these compounds it may be well to record some experiments which have been made to determine whether they lost their peculiar activity on being kept in the dark.

* A Paper read before the Royal Society, June 17, 1897.

On the 10th August last, specimens of yellow oxide, recrystallised nitrate, and chloride, the latter in solution, were each divided into two equal portions, and all placed in similar clean thin glass bottles. One sample of each was then placed in total darkness, and the other kept in the light. These samples have from time to time been tested by placing them on a photographic plate for a week and then developing the plate in the usual manner. Seven such examinations have been made at about one month's interval. No very marked difference between the samples in the light and the dark has occurred; on the whole the samples preserved in the dark have proved slightly the most active, and this was decidedly the case with all three specimens at the last examination on March 26. Another experiment was begun a little later with the black oxide of uranium, which appears to be one of the most active of the uranium compounds. Equal weights of a sample of this body were placed in two similar pill-boxes with a glass bottom; one has been kept in the dark, and the other in the light; after five months there was no difference in the effect which they produced on the photographic plate. The experiments are being continued. When repeating these different uranium experiments and using a card painted with the yellow oxide, perforated zinc was made use of simply as a screen to show the activity of the uranium compound by the density of the picture of the pattern formed; but in place of obtaining in all instances a negative of the perforated zinc, *i. e.*, the action occurring where the plate was exposed, and none where covered by the zinc, the reverse took place, and the greatest amount of action occurred underneath the zinc. This happened over and over again, and even when the experiment was varied in different ways, so that the only explanation of the action was that the zinc itself must be able to effect a change of the same kind as the uranium, at all events, to act on a photographic plate; and further experiment with zinc alone proved this to be the case; later on it became known to me that M. Colson had already described this action of zinc in a paper in the *Comptes Rendus* in January last, and had also found that similar results could be obtained with cadmium and with magnesium. He explains this remarkable action as due to vapour given off by these metals.

Both before and after seeing the account of Colson's work a large number of experiments have been made with zinc under different conditions, and there is no doubt of the ease and certainty with which the results can be obtained. The zinc, as Colson states, must be bright; if well rubbed with coarse sand-paper it is most active: probably this may, to some extent, arise from increase of surface; if cleaned with acid or with caustic alkali it is not so active, and zinc in its ordinary condition after exposure to the air ceases to be active. The salts also have no power of acting in this way. A polished piece of zinc laid on a highly sensitive photographic plate will, under certain conditions, even in four or five hours, so act on it that on development a complete picture of the zinc is produced, showing the scratches or any ruled lines or faint pattern drawn on it, or if flaws in the metal exist they are clearly seen. A slight pattern produced on zinc by pressing on it a piece of white net, and then rubbing it down with fine emery or sand-paper, will give a picture in which the pattern is very evident. In fact, such a pattern forms a satisfactory test of this action of the zinc. Very slight alterations of the surface are shown in the picture. Absolute contact of metal and plate is not necessary. If screens of different thicknesses of any inactive substance be interposed between plate and metal, thus preventing contact, the action still occurs; if the screen be very thin, a picture of the zinc surface is still obtained, but if thicker only a dark cloudy patch is formed. Still further, if a thick piece of glass tubing an inch long be placed on a photographic plate, and the upper end covered with a piece of polished zinc, in a week to a fortnight distinct action will be found to have taken place below the zinc. Since the action then is not one of mere contact,

the next point was to ascertain whether it would be transmitted through different solid or liquid media. Glass, even of the thinnest kind, was found to stop the action, but many other substances allow of its transmission. For instance, the action takes place readily through celluloid, sheet gelatin, gutta-percha tissue, collodion, vegetable parchment, real parchment, gold-beater's skin, tracing-paper, and no doubt many other bodies. With all these bodies experiments have been made by placing the medium first in contact with the zinc and the photographic plate, then by introducing a screen so as to prevent the medium from touching the zinc, and then placing a second screen so that neither zinc nor plate were in contact with the medium. The screens were made of different materials, most commonly of either white cardboard or sheet indiarubber, and of different thicknesses. The details of each experiment need not be here described; but the general results obtained are that with thin sheet gelatin, either red, green, or blue, when laid on the zinc, the action readily passes through, and a good clear picture of the surface of the zinc is obtained, and even with two sheets of gelatin a similar effect is produced. With thick sheet gelatin interposed the action on the plate still takes place, but of course the exposure must be longer. Warm solutions of gelatin were painted on polished zinc and allowed to harden; the action took place through such layers as readily as through the films. With screens used as before described to prevent contact the gelatin still allowed the action to take place through it. Thin sheets of celluloid, about 0.28 m.m. in thickness, allowed the action to take place through them, and sheets 0.81 m.m. in thickness also allowed the action to be transmitted. Again, gutta-percha tissue was found to act in the same kind of way as the gelatin and celluloid. The other media experimented with, although possibly not so uniform and continuous in structure as the foregoing, also allow this action to be transmitted to them.

Gold-beater's skin and tracing-paper both allow the action readily to pass through, and pictures of the zinc are readily obtained. If either of these bodies be placed between a piece of perforated zinc and the plate, the perforations are very distinctly shown, or if they be placed between a double screen with corresponding holes cut, a picture of the holes is readily obtained.

Both vegetable and real parchment are also transparent to this action, but not so much so as the previously-mentioned substances; the vegetable parchment is more transparent than real parchment. When in contact with the zinc, a picture of the zinc surface is obtained, but this is somewhat modified by the substance of the parchment.

If different kinds of ordinary papers, such as writing and drawing papers, be interposed between polished zinc and a photographic plate, interesting results are obtained, for the pictures formed show clearly the structure of the papers, and also show that papers have very different powers of transmitting this action. Certain writing papers are quite opaque to the action; with others, pictures of the structure and the water-mark are easily obtained.

The painting a paper with India ink does not destroy its transparency. Obviously pictures of bodies, such as skeleton leaves or dried flowers, &c., are easily obtained in this way.

A mere difference of colour does not appear to alter the absorptive power of a medium; at least, this is the case with gelatin. The thin sheets of gelatin, whether red, green, or blue, have no difference in their absorptive power, and when gelatin, coloured with aniline dyes, is painted on polished zinc, the colour does not affect the amount of action which takes place. The same thing happens if demy paper be painted with different coloured solutions of gelatin. With ordinary pigments different results are obtained, but these results need not be discussed on the present occasion.

In addition to the metals cadmium and magnesium, mentioned by M. Colson, there are certainly many others

which are able to produce effects similar to that produced by zinc. There are also certain alloys which can act in the same way. The following is a rough list of active metallic bodies approximately in the order of their activity:—Mercury, magnesium, cadmium, zinc, nickel, aluminium, pewter, fusible metal, lead, bismuth, tin, cobalt, antimony.

The above order, even if not absolutely correct, is sufficiently so to indicate that, although mercury is the most active, the other metals do not follow in the order of their fusibility or exactly according to any obvious physical property, but most nearly according to their position in the electrical series. Mercury is, then, at ordinary temperatures the most active metal, and its action is exercised not only when the photographic plate is placed half an inch or so above the metal, but when gelatin, gutta-percha, tracing-paper, vegetable parchment, are interposed. It appears, however, that the action of the mercury does not take place as readily through gelatin, but more readily through gutta-percha than is the case with zinc.

Magnesium is also a very active metal, and very good pictures, showing every scratch on its surface, is easily obtained, and also very marked effects are produced when both single and double screens are used. Cadmium also produces very good pictures, and is rather more active than zinc; nickel and aluminium are not quite so active, but give good pictures; then follow lead, bismuth, and tin. The last metal is by far the least active. Only a few alloys have at present been experimented with; brass gives no action, but ordinary pewter, and fusible metal, consisting of lead, bismuth, and tin, were found to have considerable activity, and are placed in the list between aluminium and lead. That certain alloys should act in the same way as the metals is certainly of interest, and probably of considerable importance. The oxide and sulphate, both of zinc and cadmium, were found to be devoid of any power of acting on the photographic plate. Iron, gold, and platinum are not active, and copper only very slightly. All the above results are founded on experiments in which the exposure lasted for one week; with longer exposure other metals will probably produce some action.

In order to determine whether moisture was an active agent, either directly by affecting the medium or indirectly by affecting the photographic plate, experiments were made by exposing the plates under bell-jars, in which in one case there was water, and in the other sulphuric acid or calcium chloride, and even in these extreme cases no appreciable difference was found to occur, and even if the membrane was purposely damped it did not appear to aid the action except by bringing it closer to the metal, so that aqueous vapour is not apparently an active agent in producing these reactions. In an atmosphere of hydrogen the action takes place as it does in air. Carbon dioxide, under ordinary conditions, does produce an effect, but this probably arises from its action on the zinc plate. Alteration of temperature, on the other hand, produces very marked effects; increase the temperature, and the action of the zinc is greatly increased; for instance, two similar plates, both wrapped in tinfoil and the plates separated from the zinc by means of a cardboard frame. One was placed on a water-bath and exposed to a temperature of about 70° C., and the other placed in a vessel of ice at 0° C. After five hours the one which had been exposed to the high temperature had given a black picture, while the one at the low temperature gave a picture barely visible. A similar experiment was also made with nickel, and this gave, after heating to about 70°, a good dark picture, but the corresponding experiment, when the metal was kept at 0° for five hours, gave no picture at all. Aluminium, when treated in the same way, gave at the higher temperature only a faint picture, but at the lower temperature, even after two days, no picture at all. It has already been mentioned that this action of the metals cannot pass through even thin glass, nor can it pass

through selenite, nor a layer of gum arabic, nor one of paraffin. Glass being impervious to the action, renders it somewhat difficult to try satisfactorily the action exerted by liquids, but celluloid may be used for this purpose; also mercury may be covered with a thin layer of water, and then its action entirely ceases. The action of certain salts in the dry state has, however, been tried by soaking non-glazed paper in different solutions, drying it, and then placing it, either with or without a screen, between the zinc and the photographic plate. These experiments have given some interesting results; for instance, paper soaked in the following solutions, alum, potassium chromate, zinc sulphate, and quinine sulphate, renders the paper quite opaque to the action of the zinc.

No doubt this action of alum accounts for certain papers not allowing the action to pass through them. Some singular developments of this subject have arisen from experiments made while examining the metals. A piece of polished zinc was coated with copal varnish with the object of ascertaining whether the action would take place through such a medium, and in case it did, as it was thought at the time, of demonstrating that the action could not arise from metal vapour. The experiment was quite successful; the photographic plate, notwithstanding the varnish, was strongly acted on. The experiment was repeated several times, and always with the same result; but the pictures seemed rather too good, darker than those given by the zinc alone, and on trying the copal on plain glass instead of on zinc it proved that effects apparently similar to those obtained with zinc were produced. What is known as picture copal answers very well for these experiments. That prepared by Winsor and Newton has been used. This is painted or poured on a clean, warm glass plate, and allowed to harden completely. The plate can then be used in the same way as the zinc plates. If a photographic plate be laid on the hardened varnish for two to seven days, a picture of the varnish, showing the streaks it happens to have dried in, is produced. If screens be interposed so as to prevent contact between the copal and the plate, the action still occurs, and, in fact, readily passes down a tube 1 inch long. Therefore, as with the zinc, any figure cut out in an inactive screen is readily produced on the photographic plate. Substances which are transparent or opaque to the action of the metals seem to act in the same way towards copal. It is rather more active than zinc. Glass is perfectly impervious to its action, but celluloid, gutta-percha tissue, and gelatin it permeates more readily than zinc does. The activity of the copal varies considerably under different conditions. If gum be sprinkled on a glass plate and then fused, it is not so active as when picture varnish is used. If the solid gum be dissolved in pure alcohol and ether, and applied to a glass plate as before described, it is far more active than after fusion. Heating it in a water-bath for a considerable length of time certainly deprives it of a considerable amount of its activity; but this can be revived by wetting it with ether and allowing it again to dry at ordinary temperatures. As with zinc, increase of temperature increases its activity to a great extent. Experiments similar to those with zinc were made with copal. A coated glass was exposed to a heat of about 70°, and a similar one was kept at 0°. This one after five hours gave only a faint picture, whereas the heated one gave a dark picture, and a considerable amount of action took place even through the cardboard screen. Many other bodies of the same nature as copal act in the same way. This has been proved to be the case with Damar and with Canada balsam, but copal seems to be the best representative of the class. Certain gums, such as gum arabic, gum senegal, have not the property of acting in this way. There are, however, a large number of bodies which have the power of acting in a manner similar to the copal; one of these is wood, and it possesses a very considerable amount of activity. Any ordinary smooth piece of wood laid on a photographic plate will act like zinc in impressing its picture on the plate.

A section of a young larch tree gave a good picture, showing clearly the different rings and the layer of bark, which was the darkest part of the picture. The same section, when a film of gelatin was interposed between it and the plate, still gave a good picture. Wood which is thoroughly dried and hardened is also able to act in the same way.

A piece of mahogany 3.5 m.m. thick, which had been in this form for at least thirty-five years and been carefully preserved in a dark cupboard, gave after a week's exposure a good picture, and the bottom of an old cigar-box acted equally well. Bodies such as straw, hay, bamboo, oiled silk, and, no doubt, many others, act in the same way. If wood, however, be painted with melted paraffin, it is no longer active. Ordinary charcoal also depicts itself on a photographic plate, but if it be heated for some hours in a covered crucible it loses this property. An ordinary piece of wood, if it be charred on one side by heating it with a Bunsen lamp, becomes remarkably active, as shown by placing it behind a screen with a pattern cut out. The action passes readily through different media, such as gelatin, tracing-paper, &c., vegetable parchment, &c., and the structure of the charcoal is shown, when the action has taken place, even through a sheet of vegetable parchment. Coal and coke, sulphur, sugar, on the other hand, exert no action of this kind. When trying whether a copy of a lithographic picture could be obtained by placing behind it a plate of zinc, some curious results occurred. It would seem that printer's ink in most cases is not capable of acting, like copal, on a photographic plate, but that there are many cases in which it is a remarkably active substance. Specially so is the ink used in printing many of the newspapers. The *Westminster Gazette*, for instance, is printed with an ink which very readily acts on a photographic plate. A portion of this paper with printing on only one side, laid with the blank side on the photographic plate, in a few days gives a remarkably black and distinct picture. If there be printing on both sides, then two pictures are obtained, the darker printing becoming most evident on whichever side it may be. Interpose a sheet, for instance, of gold-beater's skin, and still the picture is obtained. The *Standard* and *Daily Graphic* are also very active, and the *Times* only a little less so. The *Evening News* is only slightly active, and the *Morning Post*, *Pall Mall*, *Echo*, and *Daily News* have not the property of acting in this way; at least, those copies experimented with had not. An admission ticket to the Society of Arts laid on a photographic plate, the ink away from the plate, also gave a very distinct picture.

Another singular case of an action of this kind was met with when experimenting with the uranium salts. Not having a sufficient number of small clear glass bottles for a certain set of experiments, one of the compounds, the black oxide, was placed in a pill-box, believing that the action of the uranium would take place through the bottom of the box, and on developing the plate a dark circular space where it had stood was visible. The experiment was therefore considered very satisfactory, and, with different salts and for different objects, it was several times repeated. Ultimately it was forced upon one that the uranium salts acted more strongly when in pill-boxes than in any other way, and on placing a pill-box without any uranium salt in it on a photographic plate it was found that action had occurred, as shown by the dark circular space produced.

The experiment was repeated over and over again, with the result that most pill-boxes have the power of acting on a photographic plate. Both new and old pill-boxes from different sources were experimented with, and almost all of them found to be active. There are, however, exceptions, and these, it was noticed, were always the more expensive and elaborate boxes. On examining the structure of a pill-box it was found that it is usually made of what is known as strawboard, covered with a thin white paper; on separately testing these two materials it was apparent that the white paper was

without action on the plate, and that the strawboard was very active and produced exactly similar effects to those produced by the active pill-boxes. The inactive ones proved to be made of white cardboard, which is not an active substance. Samples of strawboard from several different sources have been tried, and all found to be active, and when separated from the photographic plate by means of screens, like the copal and the zinc, it gives a clear action. Different substances of a like nature have been tried, such as brown papers, &c. Some of them are more or less active, but none more so than common strawboard. Mr. Bevan was good enough to examine a piece of this active strawboard, but was unable to find any material other than straw present. Writing paper and, as mentioned before, white cardboard, have not this power of acting on a photographic plate, but many kinds of brown paper and no doubt many other bodies have the property. Many of the boxes in which photographic plates are packed are made of strawboard, but as the action does not pass through glass, the plates are but little or not at all acted on; but if a plate be laid face upwards in one of these boxes and left there for a week, it will be very appreciably affected. If a small piece of glass be laid on the plate, it protects the film beneath, and shows clearly the amount of action which has occurred. If a box of this kind be painted inside with melted paraffin, this action does not take place. It happened that a few months before making the above experiments others were in progress in which black net was placed on a photographic plate simply to show clearly whether the plate had been acted on, and continually a reversed picture was obtained; this at the time could not be accounted for, but now the experiment was made of simply placing the black net on the photographic plate and leaving it there for some days; then on development a clear picture of the net was produced. The action is due to some material in the black dye, for white net does not act in the same way.

The action of the vapour from a few liquids on a sensitive plate has been tried. The plate was placed about half an inch above the liquid, and a screen, with holes cut in it, was fastened against the plate. Methylated spirit acted slightly on the plate; pure alcohol and ether had no action; benzene, coal-tar, crude wood spirit, linseed oil also, had no action, but turpentine and oil of cloves produced a slight amount of action.

Such, in outline, is an account of the experiments which have already been made on this subject. One point has led on to another, and some of the results were so unexpected that the experiments had to be repeated many times before full credence could be given to them. On the present occasion it is desired to do little more than record facts; further experiments, it is hoped, may lead to explanations not now evident. The supposition that all these active substances, the metals as well as organic bodies, give off a vapour capable of acting on a photographic plate, naturally suggests itself, and that copal does give off a vapour which directly or indirectly is active there can be no doubt. At the same time, it is at least difficult to suppose that the activity of such a body as strawboard should, after the treatment it has undergone, give off at ordinary temperatures sufficient vapour to produce the effects described, and the same applies to old dry wood, &c. Still more interest attaches to the action of the metals; do they emit a vapour so delicate in constitution and in such a quantity that it can readily permeate celluloid, gelatin, &c., and produce a picture of the surface from whence it came, or is it a form of energy (possibly what has been called dark light) that these bodies emit? Zinc kept and polished in the dark loses none of its activity. An experiment has been made with the object of reflecting the zinc action from glass. This did not succeed; whether this arose from the glass not being capable of effecting such a reflection, or whether a fortnight was not sufficient time to produce in this way a visible effect, is not known, but the experiment is being

repeated. A photographic plate, suspended film upwards, over a copal plate, was acted on round the edges in the way one would imagine a vapour to act. A similar experiment is being made over a zinc plate. The action of glass proves that there is at least a marked difference between the action exerted by metallic uranium and that by zinc and other metals.

It should be stated that it is only the most sensitive photographic plates which, without extremely long exposures, give the results described. The Mawson plate has generally been used in the foregoing experiments, but the Ilford special rapid plate acts equally well, and Edwards's isochromatic snap-shot plates are particularly sensitive to the action of the uranium salts. Lumière's extra rapid are not so sensitive as the Mawson and Ilford plates, and still less sensitive are the same firm's plates for yellow and green, and for red and yellow. Other sensitive plates have not been experimented with.

ANALYSIS OF A RECENTLY DISCOVERED TOXINE SPRING.

By A. LIPP.

THE spring occurs at Seeg, near Füssen, in the Bavarian Algau district, and has the local name Marienquelle. Since 1891 it has been successfully used for medicinal purposes. It is clear and colourless and has a temperature of 7° to 8° throughout the year.

A litre of the water contains:—

	Grm.
I	0.01487
Br	0.01178
Cl	1.45660
SiO ₂	0.00650
N	0.09140
CaO	0.16050
MgO	0.06150
Fe ₂ O ₃	0.00250

Sodium, lithium, aluminium, and potassium sulphates, borates, and phosphates occur in traces.—*Berichte*, xxx., p. 309.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY
SAMPLES OF THE WATER SUPPLIED TO LONDON
FOR THE MONTH ENDING MAY 31ST, 1897.

By WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, *Metropolis Water Act*, 1871.

London, June 10th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 182 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from May 1st to May 31st inclusive. The purity of the water, in respect to organic material, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in a previous report.

In Table III. we have recorded the oxygen required to

oxidise the organic matter in all the samples submitted to analysis.

Of the 182 samples examined all were found to be clear, bright, and well filtered.

The rainfall at Oxford during the past month was 0.85 inch; the average fall for the past 30 years is 1.83 inches, showing a deficiency of 0.98 inch, reducing the total excess for the year to 1.04 inches on an actual fall of 9.67 inches. All the rain this month fell on the last five days, with the exception of 0.12 inch, which was divided between the 4th, 5th, and 11th.

The results of our bacteriological examinations of 258 samples are recorded in the following table; we have also examined 69 other samples taken from special points either at the different filter beds, or at stand-pipes:—

	Microbes per c.c.
Thames water, unfiltered (mean of 26 samples)	2937
Thames water, from the clear water wells of five Thames-derived supplies (mean of 128 samples)	40
Ditto ditto highest	233
Ditto ditto lowest	10
New River, unfiltered (mean of 26 samples) ..	388
New River, filtered (mean of 26 samples) ..	44
River Lea, unfiltered (mean of 26 samples) ..	791
River Lea, from the clear water well of the East London Water Company (mean of 24 samples)*	68

* Two additional samples abnormal.

Instead of, as formerly, examining bacteriologically each water supply once a fortnight, we are now examining samples from each water supply daily. This is therefore a much more severe test of the adequate nature of the filtration from day to day, and the mean results of our microbial estimations cannot properly compare with results obtained at periods of a fortnight apart. Further, the samples we collect from the clear water wells of the Companies are immediately cooled to the temperature of melting ice, and this temperature is maintained till the plate cultivation is commenced not later than five hours afterwards, and it is continued at 21° C., for forty-eight hours, when the colonies are counted. Such a severe examination of the process of filtration at once enables us to warn the companies if any of the filter beds are working abnormally, and already we are in a position to say that the average bacteriological quality of all the London waters has been greatly improved since the commencement of this year.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.
JAMES DEWAR.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, June 3rd, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

(Concluded from p. 296).

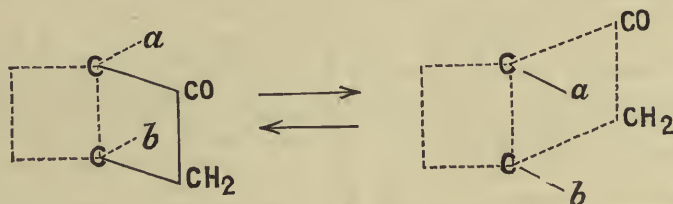
*69. "Optical Inversion of Camphor." By FREDERICK STANLEY KIPPING, Ph.D., D.Sc., and WILLIAM JACKSON POPE.

It was shown some time ago (*Trans.*, 1896, lxiii., 548) that two optically active isomeric sulphonic chlorides of the composition C₁₀H₁₅O·SO₂Cl could be obtained from the product of the action of anhydrosulphuric acid on ordinary *d*-camphor; these two compounds differed only in optical and crystallographic properties, and it was

therefore concluded that they must be regarded as optical antipodes.

This conclusion has been fully borne out by some recent work, in the course of which the properties of a considerable number of inactive compounds, prepared from inactive camphorsulphonic chloride, have been examined; it has thus been proved beyond doubt that these inactive compounds are composed of equal quantities of two enantiomorphous camphor derivatives, and that, consequently, either before or during sulphonation, the original *d*-camphor must be partly converted into its optical antipodes, *l*-camphor.

In the present paper, particular attention is drawn to this interesting case of optical inversion, as the intramolecular changes which take place appear to be entirely different from those which occur in the case of substances which contain asymmetric carbon atoms as constituents of an open chain. Although the constitution of camphor is still a matter for further investigation, it is now almost universally admitted that the skeleton of this substance consists of two closed carbon chains having two or more carbon atoms in common; further, a study of the chemical and optical properties of the various modifications of camphoric acid leads to the conclusion that each of the carboxyl groups in camphoric acid is united to an asymmetric carbon atom (Aschan, *Acta Soc. Scient.*, xxi., [5], 1-227). Admitting these two apparently well-founded assumptions, the conversion of *d*- into *l*-camphor requires that part of one of the closed chains, for example, the group $\text{—CH}_2\text{—CO—}$, should change places with the two atoms *a* and *b*, as shown in the following scheme.



As this view is based upon the two assumptions already stated, the matter is discussed from other standpoints, and it is finally concluded that the changes which occur in the optical inversion of camphor are of the nature already suggested.

*70. "Derivatives of Camphoric Acid. Part II. Optically Inactive Derivatives." By F. STANLEY KIPPING, Ph.D., D.Sc., and WILLIAM JACKSON POPE.

The optically inactive, externally compensated derivatives of camphoric acid described in this paper were prepared initially from ordinary *d*-camphor by first converting this substance into the approximately inactive camphorsulphonic chloride (*Trans.*, 1893, lxiii., 547) or camphorsulphonic bromide (*Trans.*, 1895, lxvii., 354), from which inactive π -chlorocamphor and racemic π -bromocamphor were then obtained by the method previously employed (*Trans.*, 1895, lxvii., 371).

Inactive π -chlorocamphoric acid, $\text{C}_{10}\text{H}_{15}\text{ClO}_4$, prepared by oxidising inactive π -chlorocamphor with nitric acid, crystallises in flat plates or in prisms, and melts at about $194\text{—}195^\circ$; its anhydride, $\text{C}_{10}\text{H}_{13}\text{ClO}_3$, melts at $193\text{—}194^\circ$.

Inactive π -bromocamphoric acid, $\text{C}_{10}\text{H}_{15}\text{BrO}_4$, obtained in a similar manner from racemic π -bromocamphor, is a crystalline powder melting at about $203\text{—}204^\circ$; its anhydride, $\text{C}_{10}\text{H}_{13}\text{BrO}_3$, melts at $155\text{—}156^\circ$.

These four externally compensated substances resemble the corresponding active compounds (Kipping, *Trans.*, 1896, lxix., 913; Lapworth and Kipping, *Trans.*, 1897, lxxi., 1) very closely in general properties, but the inactive anhydrides are not easily obtainable in large crystals,

active π -bromocamphoric anhydride forms large monosymmetric crystals, measurements of which are given.

Inactive *trans*- π -camphanic acid, $\text{C}_{10}\text{H}_{14}\text{O}_4$, is obtained by the decomposition of the sodium salt of π -chloro- or π -bromocamphoric acid. It crystallises from water in monosymmetric prisms, which contain one molecule of water of crystallisation, but from ethylic acetate it is deposited in anhydrous, monosymmetric, six-sided plates; the anhydrous crystals change in crystalline form at about 130° and melt at $164\text{—}165^\circ$, namely, at the same temperature as the anhydrous active acid.

Active *trans*- π -camphanic acid (*Trans.*, 1896, lxix., 929) has been further investigated, and has been found to exist in a number of different crystallographic modifications; it separates from cold water in hydrated prisms, very similar to those of the inactive acid in all respects, and it is also deposited in hydrated monosymmetric prisms from its solution in ethylic acetate; when crystallised from benzene, it affords either well-defined orthorhombic needles which contain one molecule of water of crystallisation, or a microcrystalline powder of the anhydrous acid, according to the conditions of the experiment. From a mixture of chloroform and light petroleum, it is deposited in large, transparent, anhydrous orthorhombic prisms which change in crystalline form at 100° .

The conclusions to be drawn from these and other facts bearing on the relation between the active and inactive *trans*- π -camphanic acids are discussed in the following paper.

Inactive *cis*- π -camphanic acid, $\text{C}_{10}\text{H}_{14}\text{O}_4$, is the principal product of the distillation of *trans*- π -camphanic acid. It

crystallises in large, transparent, hexagonal plates, which are indistinguishable from those of the corresponding active acid (*Trans.*, 1896, lxix., 943) except in optical behaviour; the crystals of the active acid are circularly polarising, and all of one kind, whereas those of the inactive substance show either right- or left-handed circular polarisation. It is thus possible to separate this externally compensated *cis*- π -camphanic acid into its *d*- and *l*-isomeric components.

Inactive *trans*-camphotricarboxylic acid, $\text{C}_{10}\text{H}_{14}\text{O}_6$, is obtained when inactive *trans*- π -camphanic acid is oxidised with nitric acid; it crystallises from water in lustrous, transparent, monosymmetric prisms, which differ from those of the active acid (*Trans.*, 1896, lxix., 951) deposited under similar conditions in being anhydrous, and consequently also in crystalline form. It melts at $224\text{—}225^\circ$, whereas the active acid melts at $196\text{—}197^\circ$.

Inactive *trans*-camphotricarboxylic anhydride, forms transparent monosymmetric crystals which are remarkably similar to those of the active anhydride; it melts at $253\text{—}254^\circ$ whether heated alone or with an approximately equal quantity of the active substance

*71. "Racemism and Pseudoracemism." By F. STANLEY KIPPING, Ph.D., D.Sc., and WILLIAM JACKSON POPE.

The data afforded by a comparison of the physical and crystallographic properties of the optically inactive substances described in the preceding paper with those of the corresponding active compounds (*Trans.*, 1896, lxix., 913), and a number of other facts collected during the investigation of various active and inactive π -derivatives of camphor, have led the authors to the conclusion that the pre-

sent classification of externally compensated substances into (a) mere mixtures and (b) racemic compounds, requires modification.

It has been found that optically inactive substances which are not mere mixtures of individual crystals of each of the enantiomorphous components are either very similar to, or extremely different from, their isomeric constituents in all those properties more immediately connected with crystalline structure; no intermediate degree of similarity is, in fact, observable in any case where these properties have been thoroughly examined.

Such externally compensated substances fall, therefore, into two groups. Those which closely resemble the corresponding active compounds are called *pseudoracemic*, the name racemic compound being reserved for those of the other group, of which racemic acid is the classical example.

The subdivision of optically inactive compounds has, not only an experimental, but also a theoretical basis. It can be shown that, in accordance with the present theory of crystalline structure, optically active and racemic compounds cannot assume the same type of homogeneous crystalline structure, but that an externally compensated substance may form crystalline individuals extremely similar to, but still not identical with, those of its active isomerides; in the latter case, the crystals consist of mere intercalations of those of the active modifications, and the non-identity is the result of the disturbance set up by intercalation. It is to these substances that the term *pseudoracemic* is applied.

Definitions of *pseudoracemic* and of *racemic* compounds based on these considerations are given, and some of the properties of the two classes of substances are then discussed. It is pointed out that the melting point of an externally compensated substance does not afford conclusive evidence as to its nature at ordinary temperatures, inasmuch as changes in crystalline form frequently occur with a change in temperature, and a mere mixture may become a racemic compound, and *vice versa* before the melting-point is reached; numerous experiments are quoted in support of this view. It is also concluded that solubility determinations are valueless as a means of deciding between the three classes of externally compensated substances.

The properties of a number of inactive substances described by Aschan, Emil Fischer, Liebisch, Wallach, and others are briefly discussed, and reference is made to a recent paper by Walden, which deals with the characteristics of optically active and racemic compounds.

DISCUSSION.

Dr. BONE enquired what was the practical criterion between a mixture of two optically active substances and a racemic compound proper, and whether there is any difference between the readiness with which the racemic and *pseudoracemic* forms can be resolved.

Dr. KIPPING said that, in the majority of cases, it is very difficult to distinguish between mixtures and racemic compounds except by crystallographic examination, but Liebisch's rule, that the density of a racemic compound is different from that of its optically active isomeride, if confirmed by further experimental data, might be made use of in many cases. Theoretically a racemic compound would probably be resolved into its components less readily than a *pseudoracemic* substance, but when using the methods at present known for the separation of externally compensated substances, it seems improbable that any general difference in this respect would be noticed.

*72. "Note on some New Gold Salts of the Solanaceous Alkaloids." By H. A. D. JOWETT, D.Sc.

When hyoscyne hydrobromide and auric chloride are mixed, either in concentrated, dilute, neutral, or acid solution, a red precipitate is formed which can be crystallised from a hot aqueous solution acidulated with hydrochloric acid. On analysis, the salt is found to be an additive compound of auric chloride with hyoscyne hydro-

bromide [$B \cdot HBr \cdot AuCl_3$]. When this experiment is conducted in the presence of a large excess of hydrobromic acid, a chocolate-coloured precipitate is formed which can be re-crystallised from hot dilute hydrobromic acid and forms chocolate-coloured prisms, which, on analysis, prove to be the auribromide of the base [$B \cdot HBr \cdot AuBr_3$]. Even when excess of hydrochloric acid is present the aurichloride is not formed. The analogous compounds of hyoscyamine and atropine were formed by similar reactions and resemble the corresponding salts of hyoscyne in chemical and physical properties.

Experiments were made to determine whether the bromaurichloride of formula $B \cdot HBr \cdot AuCl_3$ was an isomorphous mixture of aurichloride and auribromide, in view of the evidence adduced by Hertz (*Z. Am. C. S.*, xviii., 130) regarding the composition of the salt formed by mixing solutions of platinic chloride and potassium bromide ($K_2PtCl_4Br_2$). It was proved, however, that this view could not be adopted for the constitution of the gold salt, which must therefore be considered a true chemical compound.

*73. "Production of Camphenol from Camphor." By J. E. MARSH, M.A., and J. A. GARDNER, M.A.

The authors have described (*Trans.*, 1897, lxxi., 285) the production of an isomeride of camphor, camphenol. This substance was obtained by the action of strong sulphuric acid on chlorocamphene, $C_{10}H_{15}Cl$. Camphenol is produced by the action of the same reagent on camphene dichloride, $C_{10}H_{16}Cl_2$, which is the immediate product of the action of phosphorus pentachloride on camphor. The same camphenol is apparently produced from both the isomerides of the formula $C_{10}H_{16}Cl_2$, obtained from ordinary camphor, and a satisfactory yield is obtained in both cases. The action of strong sulphuric acid on other chloro-derivatives of terpenes has been examined. In particular, turpentine dichloride behaves in a manner very similar to the camphor derivative, but the nature of the product of the reaction has not yet been determined.

*74. "Preliminary Note on the Oxidation of Fenchene." By J. A. GARDNER and G. B. COCKBURN.

Fenchene prepared from the fenchone of fennel oil by a modification of Wallach's method was oxidised on the water-bath by moderately dilute nitric acid (1 part strong acid to 1 part water). The oxidation was complete in three days. After distilling with steam, the acid liquid was neutralised with sodium carbonate and extracted with ether, to eliminate some insoluble oily matter. The alkaline liquid was now acidified and repeatedly extracted with ether. On evaporating the ether a syrup was obtained which gradually crystallised. The crystals were purified from oily matter by washing with chloroform, and after re-crystallisation from water melted at 207° ; they proved to consist of *cis*-camphorypic acid. The oily substance, separated by chloroform, was distilled under diminished pressure. A considerable amount of decomposition took place, and an oil and a solid distilled over. The solid was crystallised from alcohol, and proved to be camphorypic anhydride (m. p. 187°).

The yield of camphorypic acid was about 9—10 per cent of the fenchene taken.

*75. "Apin and Apigenin." By A. G. PERKIN.

In a preliminary notice upon this subject (*Proc.*, 1897, xiii., 53), some derivatives and decomposition products of apigenin were described; these, together with an account of further work upon this colouring-matter, are included in the present paper. The formation of phloroglucol and parahydroxyacetophenone, as the principal products of the gentle action of alkali upon apigenin, have been confirmed, and it is now shown that, at 200° , protocathechuic acid, parahydroxybenzoic acid, and phloroglucol are obtained in the same way. These results confirm those of Gerichten (*Ber.*, 1876, ix., 1124), except as regards the production of parahydroxyacetophenone, which is not mentioned by him. On methylation, apigenin forms a

dimethyl ether, $C_{15}H_8O_3(OCH_3)_2$, yellow needles, m. p. $171-172^\circ$, which furnishes with alcoholic potash a potassium salt, decomposed by water, and with acetic anhydride a monacetyl derivative, $C_{15}H_7O_3(OCH_3)_2C_2H_3O$, colourless needles, m. p. $195-196^\circ$. The diethyl ether, $C_{15}H_8O_3(OC_2H_5)_2$, yellow needles, melts at $161-162^\circ$, and its monacetyl derivative, $C_{15}H_7O_3(OC_2H_5)_2C_2H_3O$, colourless needles, at $181-182^\circ$. As previously shown, apigenin contains three hydroxyl groups, consequently one is in the ortho-position to a carbonyl group. Decomposed with alcoholic potash, the dimethyl ether yields anisic aldehyd, anisic acid, and a phloroglucol derivative, ethylparahydroxybenzoic acid being formed from the diethyl ether under similar conditions.

These results, with the exception of the production of protocathechuic acid by means of alkali, point to a close relationship between apigenin and chrysin, $C_{15}H_{10}O_4$, the colouring-matter of poplar buds, which yields on decomposition phloroglucol, benzoic acid, and acetophenone. It is probable that apigenin is a hydroxychrysin.

This suggested relationship is borne out by the dyeing properties of the two colouring-matters, which show a close similarity. The formation of protocathechuic acid from apigenin appears to be the result of an oxidising action, for there is no evidence of a catechol nucleus in this substance. Further experiments upon its constitution are in progress.

In the previous communication (*loc. cit.*) the author erroneously assigned the discovery of the glucosoidal nature of apiin and the preparation of pure apigenin to Gerichten, instead of to Lindenhorn ("Inaug. Diss.," Würzburg, 1867).

76. "Rhamnazin." By A. G. PERKIN and H. W. MARTIN.

Rhamnazin was isolated from Persian berries by one of the authors and J. Geldard (*Trans.*, 1895, lxxvii, 496), and shown to be a quercetin-dimethylether. The present investigation was instituted to determine the position of the methoxyl groups. On methylation it yielded quercetin-tetramethylether, and from this result, and other experiments described in the paper, it evidently contains no methoxyl group in the phloroglucol nucleus in the ortho-position relatively to the carbonyl group. By fusion with alkali at 200° , rhamnazin yielded phloroglucol and protocathechuic acid, and digestion with boiling alcoholic potash gave vanillin, vanillic acid, and a non-crystalline phloroglucol derivative. Oxidised by air in alkaline solution, vanillic acid and a similar phloroglucol derivative were obtained. No free phloroglucol resulted from either of these decompositions. Taking into consideration that though the dyeing properties of rhamnazin are extremely feeble, it must still be considered a colouring-matter, these results indicate that it has the constitution of a rhamnetinmonomethylether.

77. "Experimental Verification of van't Hoff's Constant in Very Dilute Solutions." By MEYER WILDERMAN, Ph.D.

In van't Hoff's thermodynamic argument the solutions are assumed to be very dilute, and the same assumption is made in the deductions from it of Planck, Riecke, Lorentz, Boltzmann, and others. The experimental verification in dilute solutions of van't Hoff's law is therefore especially important. The freezing-point method has been worked out with greater accuracy for the purpose of this investigation (*Trans.*, 1895, lxxvii, 1; Lewis, "On the Real and Apparent Freezing-point and the Freezing-point Methods," *Proc. Roy. Soc.*, 1896; *Zeitsch. für Physik. Chemie*, 1896, xix, 233).

The author has determined van't Hoff's constant in dilute solutions with thermometers graduated to $1/1000$ th and $1/10000$ th of a degree respectively, simultaneously for a series of compounds, cane-sugar, alcohol, urea, acetone, aniline, phenol, dextrose, resorcin, maltose, milk-sugar, at converging temperatures above and below the freezing-points, using different parts of the scale of both thermometers. Small deviations only, from the theoretical

value of 1.87 , are found, due to the different sources of experimental error, van't Hoff's constant being thus confirmed in dilute solutions.

78. "The Isomeric Dibromethylenes." By THOMAS GRAY, B.Sc.

This paper contains a record of an attempt to prepare the stereo-isomeride of symmetrical dibromethylene. The following reactions are discussed:—(1) the reduction of tribromethane by sodium ethoxide; (2) the union of acetylene with bromine; (3) the reduction of acetylene tetrabromide; and (4) the addition of hydrogen bromide to bromacetylene.

By the first of these methods Tawildarow (*Ann.*, 1875, clxxvi., 22) obtained, in addition to $CH_2:CBBr_2$, a liquid boiling at 157° , and having the formula $C_2H_2Br_2$. The author confirms the observation of Michael (*Amer. Chem. Journ.*, 1883, v., 192), and finds that the only product of this reaction, under varying conditions of concentration, is $CH_2:CBBr_2$, and he attributes Tawildarow's observation to the formation of bromacetyl bromide by oxidation during the process of distillation.

The product of three other reactions is shown to be in every case the same symmetrical dibromethylene—



boiling at 110° . The author considers that the formation of this substance by the fourth method, and the probable instability of the *cis*-modification, which should result from the second reaction, point to the formula—



as representing the structure of the symmetrical dibromethylene at present known.

SOCIÉTÉ D'ENCOURAGEMENT POUR L'INDUSTRIE NATIONALE.

May 14, 1897.

President, M. MASCART.

"Art Bronze Castings." By M. MAGLIN. By using a mixture of gelatin, glycerin, and glucose, M. Maglin is able to make a complete mould of a statue in two pieces, owing to the flexibility of this substance. Perfect castings can be reproduced giving all the minute detail of the original.

M. EMILIO DAMOUR presented to the Society a laboratory form of regenerative furnace enabling temperatures of 1300° and 1400° C. to be attained. The furnace burns gas, and is made to heat muffles as large as 35 c.m. $\times$ 14 c.m. $\times$ 10 c.m. up to this high point.

May 28, 1897.

THIS meeting was also held under the presidency of M. MASCART.

The thanks of the Society were given to the various donors of books.

M. Joly was nominated a member of the Council.

A paper, accompanied by numerous experiments, was read by M. H. LE CHATELIER, on "*Grisou*," its nature, and the means of preventing accidents. This paper will be published in full in the *Bulletin*.

CHEMICAL AND METALLURGICAL SOCIETY, JOHANNESBURG.

April 17, 1897.

At the adjourned discussion on Prof. CHRISTY's paper on "*The Solution and Precipitation of Cyanide of Gold*," Mr.

BUTTERS criticised the method of precipitation proposed. He found it very difficult, he said, to make a University man realise the difficulty of carrying on, on a commercial scale, the operations which may be successfully done in the laboratory; he himself dealt with very many tons of ore per month, nearly 100,000, and he got his gold all right, but he could not succeed with this process.

Mr. L. EHLMANN then read a paper on the "*Precipitation of Gold from Cyanide Solutions*," in which he describes a series of experiments carried out by him with zinc, and zinc coated with copper, for precipitating the gold, and in every case he found that the return was higher when the zinc-copper couple was used. Hot treatment accelerated the precipitation to a very considerable degree, but the practical difficulty of heating 1000 tons of liquor a day up to 175° F. will probably prevent its adoption.

A paper on "*Liquation in Cyanide Bars*" was then read by Dr. STOCKHAUSEN. He finds that samples taken from different parts of the same bar of gold from the MacArthur cyanide process give widely different results, owing to liquation or want of homogeneity of the mass. This very unsatisfactory result is due to the neglect of stirring the molten metal before casting the ingot; dip samples and drillings also give very different results, and he comes to the conclusion that, to arrive at correct results in assaying "cyanide" gold, the only way is to take samples, after stirring, when the metal is in the molten state.

OBITUARY.

PROFESSOR FRESenius.

THE present month must be viewed as an epoch in the history of chemistry. Dr. C. Remigius Fresenius has joined the majority. To estimate what we owe to the late head of the Wiesbaden School, we need merely imagine ourselves engaged in estimating phosphoric or even sulphuric acid by the methods of the present day, as compared with those of forty years ago as laid down by experts of high and well-earned reputation. We are far from asserting that all the niceties and improvements which have been latterly introduced into the practice of the laboratory are due to the work of Fresenius; but we know to what an extent he has been, year by year, our guiding soul. He and his pupils have taught us how to find, in a specimen submitted for examination, what really exists, and—what is certainly in many cases not less important—to avoid finding what is not present, and thus to escape founding erroneous theories.

Still we must gratefully own that Fresenius had to a great extent completed his work. For the last few years his labours had mainly ceased. His sons, Dr. Heinrich Fresenius, Dr. W. Fresenius, and his son-in-law, Dr. Ernst Hintz, have been carefully trained to follow in his steps, and we feel confident that the Wiesbaden School of Chemistry will continue to do good and useful work. To the best of our knowledge the *Zeitschrift für Analytische Chemie* will continue to enjoy the co-operation of the numerous able chemists who have heretofore contributed to its pages.

NOTICES OF BOOKS.

City and Guilds of London Institute for the Advancement of Chemical Education. Report to the Governors. March, 1897. Gresham College, Basinghall Street, London, E.C.

THIS Report is minute, and on the whole satisfactory.

The constitution of the Institution is somewhat complex. There is a list of Governors, including the

President (H.R.H. the Prince of Wales); a body of Vice-Presidents, some of whom hold their position *ex officio*, such as the Presidents of the Royal and the Chemical Societies and the Chairman of the Society of Arts, a number nominated or appointed by the Corporation and by the principal Companies. As the nominees or representatives of the Goldsmiths' Company, we notice the names of Mr. G. Matthey, F.R.S., F.C.S.; Sir F. Bramwell, F.R.S.; Sir Frederick Abel, Bart., K.C.B., D.C.L., F.R.S.; and many other gentlemen widely known in scientific and technical circles.

The Council is a distinct body, though including not a few of the same names.

Then follow the Executive Committee, in which we meet—we may say welcome—not a few of the members of the former bodies.

Lastly, comes the Staff of the Institute's Colleges, including, on behalf of the Central Technical College, Prof. H. E. Armstrong, Ph.D., F.R.S.; F. S. Kipping, Ph.D., D.Sc., Assistant at the Research Laboratory; G. S. Moody, D.Sc.; and W. L. Pope, Instructor in Crystallography.

In the department of applied chemistry we find Prof. Raphael Meldola, F.R.S., F.C.S., beside three assistants.

It does not appear with quite sufficient clearness what are the duties or functions of these Boards.

It appears that on April 22nd, 1896, a communication was received from the Mercers' Company requesting the Governors to appoint a Special Committee to inquire into the expenditure of the Central Technical College of the Institute, with especial reference to the results attained. The Special Committee appointed consisted of the Lord Chancellor, six engineers, six chemists (using the latter term in the acceptance which it bears in France, Germany, &c.), four members of the Technical Education Board of the London County Council, one member of the last Royal University Commission, and twelve others. The results of the Report were very satisfactory. The conclusion was that the work in the College had been eminently successful and fully commensurate with the expenditure, and that its objects are well deserving of every support and encouragement that the Corporation and City Companies of London can give them.

The various Companies and other bodies who support the Institute have distinctly expressed themselves satisfied with the work as carried on. The gross cost per student for the years 1894 and 1895 has been £54, whilst the cost per student at the Zürich Polytechnic is about £59; at the American Institutions from £60 to £63, and at the Royal College of Science about £67.

Alumni Report of the Philadelphia College of Pharmacy.

May, 1897, vol. xxxiii., No. 8.

THE contents of this number begin with a short biographical sketch of the *honour* students of 1897. Then follows a brief account of the sixth Pharmaceutical Meeting of the Session 1896-1897, at which there were discussions on ointment of mercuric nitrate, the analysis of "Gelsemium," and a contribution on "The Presence of Starch and Strontium Sulphate in Opium, and their Influence on Assaying," in which we find the curious statement that, though starch has been often found in opium in quantities varying from a trace to 8 per cent, it should not be considered as an adulterant.

The rest of the number contains accounts of the annual meeting, reception, &c., and a list of the graduates of the year.

A Text-book of Chemistry. Translated into Chinese by JAS. B. NEAL, M.D.

WE have received from the English author, Prof. Clowes, a most interesting publication, hailing from the Flowery Land. It is a text-book of chemistry, printed throughout

in Chinese characters, being for the most part a translation of Clowes's "Analytical Chemistry," the only exception being the third chapter, which has been taken mostly from Fownes's "Manual of Chemistry." The book is intended simply for laboratory use. It is the translator's firm conviction that only by practical work in the laboratory can a knowledge of chemistry be gained, which will be more than a mere learning by rote, and especially so in the case of Chinese students.

The elements are, with four exceptions,—viz., potassium, calcium, zinc, and arsenic—called by the names or characters adopted by Dr. Fryer: these four exceptions are so frequently met with in Chinese medical works that it was thought best to adhere to the terms used by Dr. Kerr, to prevent the confusion which would inevitably arise through the use of two sets of names.

CORRESPONDENCE.

THE DISCOVERY OF OXYGEN AND THE "ENCYCLOPÆDIA BRITANNICA."

To the Editor of the Chemical News.

SIR,—In spite of the indisputable fact that oxygen gas was first isolated and recognised as a supporter of combustion by the Rev. Dr. Joseph Priestley, on the 1st of August, 1774, French writers on the history of chemistry have persistently claimed the honour of this discovery for Lavoisier. This unhappy attempt to transfer credit from an English man of science to a French one would not have received the support of Lavoisier himself, whose immortality in the annals of chemistry does not need to be magnified by mendacious claims.

I have grown accustomed to this perversity on the part of the French; but imagine my surprise when I found that peculiarly English work the "Encyclopædia Britannica" coolly perpetuating this inaccuracy for the benefit of its Anglo-Saxon readers. In the ninth edition, under the caption "Alchemy" (vol. i., 1878), are the following paragraphs:—

"Lavoisier, who by discovering oxygen destroyed the theory of Stahl."

"... and finally in disengaging from the red-oxide of mercury oxygen gas, that Proteus which so often eluded the grasp of the alchemists, till at last it was held fast by the subtle analysis of Lavoisier" (page 462).

After I had recovered from the shock at seeing these statements in that monument of English erudition, I turned to the end of the article on alchemy to ascertain the authorship; it is signed "J. A.," which the key at the close of the volume interprets "Jules Andrieu." *Hinc illæ lacrymæ!* Why the Editor-in-chief sought for a Frenchman to write the article in question it is difficult to conceive. Were there no Englishmen in 1878 competent to compile such an essay? And, having secured the article, were there no members of the editorial staff who had heard of Priestley and his discoveries in chemistry? Unfortunately for the staff, the articles "Chemistry" and "Priestley," in which the fact is correctly given, only appeared in later volumes.

Those curious in the matter will find the whole article on alchemy of interest, but not because it is judiciously written.—I am, &c.,

H. C. B.

ESTIMATION OF CARBON IN FERRO-CHROME.

To the Editor of the Chemical News.

SIR,—A complete and accurate analysis of ferro-chrome is not an easy thing to do. I am pleased, therefore, to notice Mr. Saniter's letter, because he has long been interested in the matter. The letter, however, indicates that he has confused the preliminary experiments and

temporary method with the procedure finally adopted, viz., combustion with lead peroxide.

On page 243, col. 1 (CHEMICAL NEWS, vol. lxxv.) it is stated that "the mixture [PbO₂ and ferro-chrome] fuses, and there is the same need to quicken the oxygen to supply the absorption in the boat, but all else goes on with comparative quietness and at ordinary temperatures." The covered boat, previously mentioned, is necessary only with the lead chromate or copper oxide at the higher temperatures.

I have just finished a duplicate determination on 1.00 and 1.25 grms. respectively, and, on unwrapping the asbestos from the glass tube, find only a faint deposit immediately over the boat. With such large amounts of peroxide and ferro-chrome the reaction is, of course, more vigorous than it would be with Mr. Saniter's proportions. That his assays do not spurt causes no surprise, and one may justifiably suspect that the copper oxide is not greatly concerned in the matter when, under less favourable circumstances, assays do not spurt without it. The copper oxide may preserve the porcelain from the attack of the litharge by diluting it, as it were; but this is a very minor point: only the boat can be attacked, and with the peroxide alone we use them over and over again after re-heating in the muffle and pouring away the melt.

The complication then narrows itself down to this:—Mr. Saniter uses a mixture of copper oxide and litharge, and I use lead peroxide alone.

It should be noticed that Mr. Saniter's ferro-chrome must needs be ground fine in an agate mortar—a labour he has elsewhere endeavoured to avoid. It may be seen (p. 243, col. 2) that PbO₂ much more readily than CuO—or presumably than a mixture of CuO and PbO—attacks the ferro-chrome. In this respect, then, it would be less troublesome to use the single reagent.

The comparative advantage of using litharge or the peroxide would favour the former if—to use Mr. Saniter's expression—it were "free from substances which might be given off and absorbed by the potash." So far we have found that, having once determined the blank of the peroxide, it may be relied upon to remain unchanged. The reaction $2\text{PbO}_2 = 2\text{PbO} + \text{O}_2$ seems to be nearly, if not altogether, completed before the subsequent reaction, $2\text{PbO} + \text{C} = \text{CO}_2 + 2\text{Pb}$,* is begun. It would be a slight advantage, then, if PbO could be used straightaway, but unfortunately PbO—perhaps the litharge variety in a less degree—absorbs CO₂ from the air, and particularly so if damp. It would be necessary, therefore, on using this reagent, to prepare it immediately before use, and to take the troublesome precaution to keep it perfectly dry, and practically out of contact with the air. Thus the complications, if great accuracy is a vital consideration, would seem to hamper the use of the mixed reagents.

I ought to state that I have not largely experimented with litharge. The previous considerations, founded on known properties of lead protoxide, and the fact that the peroxide as received from the dealers gave a small constant blank, led me to prefer the latter.—I am, &c.,

H. BREARLEY.

June 14, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 21, May 24, 1897.

Tools and Arms of the Age of Pure Copper in Egypt.—Prof. Berthelot.—The author gives a list of antique

* The third reaction in the tube, which explains the need to quicken the oxygen, may be written $2\text{Pb} + \text{O}_2 = 2\text{PbO}$. An attempt is being made to substantiate these equations by direct experiment.—H. B.

articles sent him by M. de Morgon. They belong to the most remote ages of the Egyptian empire. They all consist of copper nearly pure, sometimes containing arsenic, but no tin, lead, or zinc. They were then succeeded by bronze and then by iron.

On various Liquids contained in Antique Vases.—Prof. Berthelot.—Two of these liquids have been forwarded to the author by Th. Hubert, custos of the Archæological and Ceramic Museum of the city of Reims; they seem to belong to the Gallo-Roman epoch. They have been extracted from a glass bottle, and consisted of an aqueous liquid covered by fatty matter, chiefly liquefied. The solid portion consisted of stearic and palmitic acids accompanied by a neutral fatty matter. The aqueous matter consists of a mixture of volatile acids with watery vapour ($C_nH_{2n}O_2$), and of fixed acids ($C_nH_{2n-2}O_4$), mixed with acids ($C_nH_{2n-2}O_3$?). A perfectly distinct specimen was obtained from a Syrian tomb. It was merely water with small quantities of calcium bicarbonate chloride and traces of nitrates. It is apparently water of infiltration. Observers should mistrust the accidental introduction of water by infiltration into antique vessels.

Action of Light upon Gaseous Mixtures, especially Mixtures of Chlorine and Hydrogen.—Armand Gautier and H. Helier.

On the Sojourn of General Poncelet at Saratow.—Germain Babst.—This paper, though placed under the heading "History of the Sciences," is merely a fragment of the history of the retreat of the Grande Armée.

New Improvement of the Grisometer.—M. Grehont.—The new model is constructed of stout glass cylinder, with two concentric plates of sheet nickel. It is charged with soda-lye free from Cl at about 15 per cent NaOH. The silver-voltmeter is universally recognised as the most accurate voltmeter.

The External Surface of Cast-iron Raised to a Red Heat Converts Carbonic Acid into Carbon Monoxide.—M. Grehont.—The author infers from his researches that we must abandon heating rooms by walls of cast metal heated to redness.

Reflection of Light by a Long and Narrow Surface.—M. Gony.—MM. Nichols and Rubens, when operating with thermic rays of great wave-length (24μ) have found that a long narrow band of silver reflects these radiations whilst polarising them perpendicularly to the length of the band, whence there results an interesting approximation to electric waves.

An Antianodic Phosphorescent System, and on the Anodic Rays.—C. Maltézos.—The phenomena observed indicate the existence under certain conditions of anodic rays which provoke the phosphorescence of glass, visible and invisible, and which most generally are diffused or which do not reach the glass.

Properties of certain Radiations of the Spectrum.—Gustave Le Bon.—A reply to the paper by Prof. Becquerel, and his objections to his hypothesis of "black rays."

Precipitation of Zinc Sulphide for the Determination of that Metal.—J. Meunier.—All chemists who have been concerned themselves with the determination of zinc know the difficulties of collecting on a filter zinc sulphide precipitated by ammonium sulphide. The filtrate is turbid and the filtration is soon stopped. To the solution of zinc, which is properly luke-warm, he adds ammonia, and when the precipitate of zinc oxide is formed he continues to add this reagent, but cautiously, and passing in sulphuretted hydrogen bubble by bubble, ceasing as soon as the precipitation of the zinc is complete.

Remarks on the Formation-heat of Sodac Acetylene.—M. de Forcrand.—A thermo-chemical paper, not suitable for abstraction.

New Compounds of Pyridine, Piperidine, and Quinoline with the Metallic Salts.—Raoul Varet.—

The compounds described are pyridine, bromocypripine, iodozincate, cyanozincate, bromocadmiate, and bromonickelate.

Solubility of Ecgonin.—Oechsner de Coninck.—The author gives the solubilities of ecgonin in 25 solvents.

Comparative Study of the Quotients of Acids and of Fermentation observed during the Ripening of Fruits.—C. Gerber.

Denaturation of Alcohol.—Ernest Barillot.

MISCELLANEOUS.

The Philadelphia Museums.—This institution is in a measure similar to the Imperial Institute. It is divided into several departments—that of Foreign Manufactures has for its object the showing to the American manufacturers what their foreign competitors are exporting. There is a large collection of all kinds of goods, each sample being accompanied by the manufacturer's price. One great innovation which we should be very sorry to see introduced into England, is the analysis and reporting on of all raw products sent, entirely free of charge, the Institution being supported by the public funds. We hope the city also helps to support the analytical chemists who must be adversely affected by this action on its part.

Inauguration of the Monument Erected to the Memory of Jean Servais Stas.—After the death of Stas an international commission was constituted, and an appeal was made to the scientific men of all countries, with the object of perpetuating the name and works of this illustrious chemist. With the funds collected the committee have already raised one imperishable monument to his memory—a complete edition of his works. But the three volumes of which this consists appeal more or less to the *élite*; so it was decided that a monument of this man, who by his genius rose from the ranks, should be erected, accessible to all. J. S. Stas first studied at the Faculté de Médecine at the University of Louvain, and from there went to Dumas' laboratory. It was here that, under the direction and with the collaboration of his illustrious master, he made his researches on the atomic weight of carbon, and wrote his "Mémoire sur les types chimiques." Some years afterwards he was appointed professor of chemistry at the Military School at Brussels, but he worked under great difficulties. The Government refusing even to complete the laboratory, Stas was constrained to defray the expenses out of his own pocket. In 1860 he wrote to an intimate friend: "To continue my researches I have to make such sacrifices that I am reduced to a condition bordering on poverty." He was offered help by Liebig, but was too patriotic to accept it from Germany, till at last his own Government, recognising the value of his work, made him a grant of 6000 francs, and it is to him and his persistent efforts that the present position of Science in Belgium is due. We cannot now follow him through his long and great career; by his nobility of character, his rare intellect and intelligence he inspired affection and commanded respect from all who knew him, and his work, better than empty titles, will keep his memory green.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Marking Inks.—I should be greatly obliged for information as to whether there is a chance of obtaining a book, or one or a series of reports upon the subject relating to manufacture of the latest marking inks, especially vegetable ones. There are one or two societies in France dealing with the subject, but I do not know whether they issue any reports or books on that subject; if so, I should be glad to learn how they are to be obtained.—SULPHO.

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DIAMONDS.*

By WILLIAM CROOKES, F.R.S., M.R.I.

(Continued from vol. lxxv., p. 302).

Depositing Floors.

OWING to the refractory character of blue ground fresh from the mines, it has to be exposed to atmospheric influences before it will pulverise under the action of water and mechanical treatment. It is brought to the surface and spread on the floors. Soon the heat of the sun and moisture produce a wonderful effect. Boulders, hard as ordinary sandstone when fresh from the mine, commence to crumble. At this stage the treatment of the diamonds assumes more the nature of farming than mining. To assist pulverisation by exposing the larger pieces to atmospheric influences, the ground is frequently harrowed and occasionally watered. The length of time necessary for crumbling the ground preparatory to washing, depends on the season of the year and the amount of rain. The longer the ground remains exposed the better it is for washing. When the process is complete the softened friable blue clay is again loaded into trucks and taken to the washing machinery, where it is agitated with water and forced through a series of revolving cylinders perforated with holes about an inch in diameter; incorrigible lumps that will not pass the cylinders are again subjected either to the weathering process or passed between crushing rollers.

Washing and Concentrating Machinery.

The fine ground which has passed through the holes in the cylinder, together with a plentiful current of water, flows into the washing pans. These pans are of iron, 14 feet in diameter, furnished with ten arms each having six or seven teeth. The teeth are set to form a spiral, so that when the arms revolve the teeth carry the heavy deposit to the outer rim of the pan, while the lighter material passes towards the centre and is carried from the pan by the flow of water. The heavy deposit contains the diamonds. It remains on the bottom of the pan and near its outer rim. This deposit is drawn off every twelve hours by means of a broad slot in the bottom of the pan. The average quantity of blue ground passed through each pan is from 400 to 450 loads in ten hours. The deposit left in each pan after putting through the above number of loads amounts to three or four loads, which go to the pulsator for further concentration.

The Pulsator.

The Pulsator is an ingeniously designed, somewhat complicated machine for dealing with the diamantiferous gravel already reduced one hundred times from the blue ground; the pulsator still further concentrating it till the stones can be picked out by hand. The value of the diamonds in a load of original blue ground is about 30s., the gravel sent to the pulsator from the pans, reduced a hundred-fold, is worth £150 a load.

The sorting room in the pulsator house is long, narrow, and well lighted. Here the rich gravel is brought in wet, a sieve full at a time, and is dumped in a heap on tables covered with iron plates. The tables at one end take the coarsest lumps, next comes the gravel which passed the $\frac{3}{8}$ -inch holes, then the next in order, and so on. The first sorting, where the danger of robbery is greatest, is done by thoroughly trustworthy white men. Sweeping the heap of gravel to the right, the sorter scrapes a little of it to the centre of the table by means of a flat piece of sheet zinc. With this tool he rapidly surveys the grains, seizes the diamonds, and puts them into a little tin box in front of him. The stuff is then swept off to the left, and another lot taken, and so on, till the sieve-ful of gravel is exhausted, and another brought in.

The diamond has a peculiar lustre, impossible to mistake. On the sorting table the stones look like clear pieces of gum arabic, but with an intrinsic lustre which makes a conspicuous shine among the other stones.

Watching the white men in the sorting room is an experience but tame compared to the excitement of taking a sorter's place at the big diamond table and disinterring from the gravel diamonds usually described as the finest and biggest found for many a day. The interest, however, abates when the amateur sorter is told that the jewels may not be carried away as mementos!

Sometimes as many as 8000 carats of diamonds are separated in one day, representing about £10,000 in value.

Diamonds occur in all shades, from deep yellow to pure white and jet-black, from deep brown to light cinnamon; they are also green, blue, pink, yellow, orange, and opaque.

The Diamond Office.

From the pulsator sorting room the stones are taken to the Diamond Office to be cleaned in acids and sorted into classes by the valuers, according to colour and purity. It is a sight for Aladdin to see the valuers at work in the strong-room of the De Beer's Company at Kimberley. The tables are literally heaped with stones won from the rough blue ground,—stones of all sizes, purified, flashing and of inestimable price; stones that will be coveted by men and women all the world over; and

* A Lecture delivered at the Royal Institution, Friday, June 11th, 1897.

last but not least stones that are probably destined to largely influence the development and history of a whole huge continent.

When the diamantiferous gravel has been washed down to a point at which the stones can be picked out by hand, a good plan for separating them is by their specific gravities. The following table gives the specific gravities of the minerals found on the sorting tables. I have also included the specific gravities of two useful liquids:—

	Specific gravity.
Hard graphite	2.5
Quartzite and granite	2.6
Beryl	2.7
Mica	2.8
Hornblende	3.0
METHYLENE IODIDE	3.3
DIAMOND	3.5
THALLIUM LEAD ACETATE	3.6
Garnet	3.7
Corundum	3.9
Zircon	4.4
Barytes	4.5
Chrome and titanite iron ore	4.7
Magnetite	5.0

This table shows that if I throw the whole mixture of minerals into methylene iodide, the hornblende and all above that mineral will rise to the surface; while the diamond and all minerals below will sink to the bottom. If I now take these heavy minerals, and throw them into thallium lead acetate, they will all sink except the diamond which floats and can be skimmed off.

In illustration, I have arranged an experiment. In front of the lantern is a cell containing a dense liquid; when I throw into it several minerals of different specific gravities, some sink whilst others swim, and these swimmers can easily be skimmed from the surface.

The "Compound" System.

With gems like diamonds, where infinite riches are concentrated in so small a bulk, it is not surprising that safeguards against robbery are elaborate. The Illicit Diamond Buying (I.D.B.) laws are stringent, and the searching, rendered easy by the "compounding" of the natives, is of a drastic character. In fact, it is very difficult for a native employé to steal diamonds; even were he to succeed, it would be almost impossible to dispose of them, as a potential buyer would prefer to secure the safe reward for detecting a theft rather than run the serious risk of doing convict work on the Cape Town Breakwater for a couple of years. Before the passing of the "Diamond Trade Act" the value of stolen diamonds reached nearly one million sterling per annum.

One great safeguard against robbery is the "compound" system of looking after the natives. A "compound" is a large square, about 20 acres in extent, surrounded by rows of one-storey buildings of corrugated iron. These are divided into rooms each holding about twenty natives. A high iron fence is erected around the compound, 10 feet from the buildings. Within the enclosure is a store where the necessities of life are supplied to the natives at a reduced price, and wood and water free of charge. In the middle is a large swimming-bath with fresh water running through it. The rest of the space is devoted to games, dances, concerts, and any other amusement the native mind can desire. In case of accident or illness there is a well-appointed hospital where the sick are tended. Medical supervision, nurses, and food are supplied free by the Company.

As a rule the better class of natives—the Zulus, Matabeles, Basutos, Bechuanas—when well treated, are honest and loyal.

In the compound are to be seen representatives of nearly all the picked types of African tribes. Each tribe keeps to itself, and to go round the buildings skirting the

compound is an admirable object-lesson in ethnology. At one point is a group of Zulus; next we come to Fingoes; then Basutos; beyond come Matabele, Bechuanas, Pondos, Shangains, Swazis, and other less-known tribes, each forming a distinct group, or wandering around making friendly calls. We went one afternoon to the De Beers compound when most of the natives were assembled, and having a camera with me I was naturally glad to get as many photographs as I could. I have to thank Captain Dallas, Mr. Moses, and Mr. Mandy, the Superintendents of the respective compounds, who speak all the dialects fluently, for their kindness in showing us round and improvising dances and concerts, for the benefit of my camera.

The clothing in the compound is diverse and original. Some of the men are great dandies, whilst others think that in so hot a climate a bright coloured pocket-handkerchief or "a pair of spectacles and a smile" is as great a compliance with the requirements of civilisation as can be expected.

The Diamond.

So distinctive are the characters in diamonds from each mine that an experienced buyer at once tells the locality of any particular parcel of stones. De Beers and Kimberley mines are distinguished by large yellowish crystals. Dutoitspan yields mainly coloured stones, while Bullfontein—half a mile off—produces small white stones, occasionally speckled and flawed, but rarely coloured. Diamonds from the Wesseltion mine are nearly all irregular in shape, a perfect crystal is rare, and most of the stones are white, few yellow. Diamonds from the Leicester mine have a frosted, etched appearance; they are white, the crystallisation irregular ("cross-grained"), and they are very hard. The newly discovered "Newlands" mines in Griqualand West are remarkable for the whiteness of their diamonds and for their many perfect octahedral crystals. Jagersfontein stones, in the Orange Free State, take the prize for purity of colour and brilliancy, and they show that so-called "steely" lustre characteristic of old Indian gems. Stones from Jagersfontein are worth nearly double those from Kimberley and De Beers.

Monster diamonds are not so uncommon as is generally supposed. Diamonds weighing over an ounce (151.5 carats) are not uninfrequent at Kimberley, and there would be no difficulty in getting together a hundred of them. Not long ago, in one parcel of stones at the office of Wernher, Beit, and Co., I saw eight perfect crystals, each over an ounce and one that weighed two ounces. The largest known diamond—a true mountain of light—weighs 970 carats, over half a pound. It was found four years ago at Jagersfontein. It is perfection in colour, but has a small black spot in the centre. Diamonds smaller than a small fraction of a grain elude the sorters and are lost. A microscopic examination of blue ground from Kimberley, after treatment with appropriate solvents, shows the presence of microscopic diamonds, white, coloured, and black, also of boart and carbonado.

From two to three million carats of diamonds are turned out of the Kimberley mines in a year, and as five million carats go to the ton, this represents half a ton of diamonds. To the end of 1892, ten tons of diamonds had come from these mines, valued at £60,000,000 sterling. This mass of blazing diamonds could be accommodated in a box five feet square and six feet high.

The diamond is a luxury for which there is only a limited demand. From 4 to 4½ millions sterling is as much as is spent annually in diamonds; if production is not regulated by demand, there will be over-production, and the trade will suffer. By regulating the output, since the consolidation in 1888 the directors have succeeded in maintaining prices.

Outside companies and individuals collect diamonds to the value of about a million annually.

Graphite.

Intermediate between soft carbon and diamond come

the graphites. The name graphite is given to a variety of carbon, generally crystalline, which in an oxidising mixture of chlorate of potassium and nitric acid forms graphitic acid easy to recognise. Graphites are of varying densities, from 2.0 to 3.0, and generally of crystalline aspect. Graphite and diamond pass insensibly into one another. Hard graphite and soft diamond are near the same specific gravity. The difference appears to be one of pressure at the time of formation.

Some forms of graphite exhibit a remarkable property, by which it is possible to ascertain approximately the temperature at which graphites were formed, or to which they have subsequently been exposed. Graphites are divided into "sprouting" and "non-sprouting." When obtained by simple elevation of temperature in the arc or the electric furnace they do not sprout; but when they are formed by dissolving carbon in a metal at a high temperature and then allowing the graphite to separate out on cooling, the sprouting variety is formed. One of the best varieties is that which can be separated from platinum in ebullition in a carbon crucible. The phenomenon of sprouting is easily shown. I place a few grains in a test-tube and heat it to about 170° C., when as you see it increases enormously in bulk and fills the tube with a light form of amorphous carbon.

The resistance of a graphite to oxidising agents is greater the higher the temperature to which it has previously been exposed. Graphites which are easily attacked by a mixture of fuming nitric acid and potassium chlorate are rendered more resistant by strong heat in the electric furnace.

I will now briefly survey the chief chemical and physical characteristics of the diamond, showing you by the way a few experiments that bear upon the subject.

Combustion of the Diamond.

When heated in air or oxygen to a temperature varying from 760° to 875° C. according to its hardness, the diamond burns with production of carbonic acid. It leaves an extremely light ash, sometimes retaining the shape of the crystal, consisting of iron, lime, magnesia, silica, and titanium. In boart and carbonado the amount of ash sometimes rises to 4 per cent but in clear crystallised diamonds it is seldom higher than 0.05 per cent. By far the largest constituent of the ash is iron.

The following table shows the temperature of combustion in oxygen of different kinds of carbon:—

	°C.
Condensed vapour of carbon	650
Carbon from sugar, heated in an electrical furnace	660
Artificial graphites, generally	660
Graphite from ordinary cast-iron	670
Carbon from blue ground, of an ochrey colour	690
" " " very hard and black	710
Diamond, soft Brazilian	760
" " hard Kimberley	780
Boart from Brazil	790
" " from Kimberley	790
" " very hard, impossible to cut	900

At the risk of repeating an experiment shown so well at this table by Professor Dewar, I will heat a diamond to a high temperature in the oxyhydrogen blowpipe and then suddenly throw it in a vessel of liquid oxygen. Notice the brilliant light of its combustion. I want you more especially to observe the white opaque deposit forming in the liquid oxygen. This deposit is solid carbonic acid produced by the combustion of the carbon. I will lead it through baryta water, and you will see a white precipitate of barium carbonate. With a little more care than is possible in a lecture I could perform this experiment quantitatively, leading the carbonic acid and oxygen, as they assume the gaseous state, through baryta-water, weighing the carbonate so formed, and showing that one gramme of diamond would yield 3.666 grammes

of carbonic acid—the theoretical proportion for pure carbon.

Some crystals of diamonds have their surfaces beautifully marked with equilateral triangles, interlaced and of varying sizes. Under the microscope these markings appear as shallow depressions sharply cut out of the surrounding surface, and these depressions were supposed by Gustav Rose to indicate the probability that the diamonds at some previous time had been exposed to incipient combustion. Rose also noted that striations appeared on the surfaces of diamonds burnt before the blowpipe. This experiment I have repeated on a clear smooth diamond, and have satisfied myself that during combustion in the field of a microscope, before the blowpipe, the surface becomes etched with markings very different in character from those naturally inscribed on crystals. The artificial striæ are cubical and closer massed, looking as if the diamond during combustion had been dissected into rectangular flakes, while the markings natural to crystals appear as if produced by the crystallising force as they were being built up.

I exhibit on a diagram a form of graphite from the Kimberley blue ground (reproduced from M. Moissan's work) which in its flaky crystalline appearance strangely resembles the surface of a diamond whose internal structure has been partially dissected and bared by combustion. It looks as if this piece of graphite was ready to separate out of its solvent as diamond, but owing to some insufficient factor it retained its graphitic form.

Physics of the Diamond.

The specific gravity of the diamond is from 3.514 to 3.518. For comparison, I give in tabular form the specific gravities of the different varieties of carbon:—

Amorphous carbon	1.45	1.70
Graphite	2.11	3.0
Hard gas coke	2.356	
Boart	3.47	3.49
Carbonado	3.50	
Diamond	3.514	3.518

The diamond belongs to the isometric system of crystallography. It frequently occurs with curved faces and edges. Twin crystals (macles) are not uncommon. Having no double refraction it should not act on polarised light. But as is well known, if a transparent body which does not so act is submitted to strain of an irregular character it becomes doubly refracting, and in the polariscope reveals the existence of the strain by brilliant colours arranged in a more or less defined pattern according to the state of tension in which the crystal exists. Under polarised light I have examined many hundred diamond crystals, and with few exceptions all show the presence of internal tension. On rotating the polariser, the black cross, which is most frequently seen, revolves round a particular point in the inside of the crystal, and on examining this point with a high power, we see sometimes a slight flaw, more rarely a minute cavity. The cavity is filled with gas at an enormous pressure, and the strain is set up in the stone by the effort of the gas to escape.

It is not uncommon for a diamond to explode soon after it reaches the surface, and some have been known to burst in the pockets of the miners or when held in the warm hand. Large crystals are more liable to burst than smaller pieces. Valuable stones have been destroyed in this way, and it is whispered that cunning dealers are not averse to allowing responsible clients to handle or carry in their warm pockets large crystals fresh from the mine. By way of safeguard against explosion, some dealers imbed large diamonds in raw potato to insure safe transit to England.

I will project some diamonds on the screen by means of the polarising microscope, and you will see by the colours how great is the strain to which some of them are exposed.

In the substance of many diamonds we find enclosed black uncrystallised particles of graphite. There also occur what may be considered intermediate forms between the well-crystallised diamond and graphite. These are "boart" and "carbonado." Boart is an imperfectly crystallised diamond, having no clear portions, therefore it is useless for gems. Boart is frequently found in spherical globules, and may be of all colours. It is so hard that it is used in rock drilling, and when crushed it is employed for cutting and polishing other stones. Carbonado is the Brazilian term for a still less perfectly crystallised form of carbon. It is equally hard, and occurs in porous masses, and in massive black pebbles, sometimes weighing a couple or more ounces.

Diamonds vary considerably in hardness, and even different parts of the same crystal are decidedly different in their resistance to cutting and grinding. The famous Koh-i-noor, when cut into its present form, showed a notable variation in hardness. In cutting one of the facets near a yellow flaw, the crystal became harder and harder the further it was cut into, until, after working the mill for six hours at the usual speed of 2400 revolutions a minute, little impression was made. The speed was according increased to more than 3000, when the work slowly proceeded. Other portions of the stone were found to be comparatively soft, and became harder as the outside was cut away.

Beautifully white diamonds have been found at Inverel, New South Wales, and from the rich yield of the mine and the white colour of the stones, great things were expected. A parcel of many hundred carats came to England, when it was found that they were so hard as to be practically unworkable as gems, and I believe they were ultimately sold for rock boring purposes.

I will illustrate the intense hardness of the diamond by an experiment. I place a diamond on the flattened apex of a conical block of steel, and on the diamond I bring down a second cone of steel. With the electric lantern I will project an image of the diamond and steel faces on the screen, and force them together by hydraulic power. Unless I happen to have selected a diamond with a flaw, I shall squeeze the stone right into the steel blocks without injuring it in the slightest degree.

But it is not the hardness of the diamond so much as its optical qualities that make it so highly prized. It is one of the most refracting substances in Nature, and it also has the highest reflecting properties. In the cutting of diamonds advantage is taken of these qualities. When cut as a brilliant the facets on the lower side are inclined so that light falls on them at an angle of $24^{\circ} 13'$, at which angle all the incident light is totally reflected. A well-cut diamond should appear opaque by transmitted light except at a small spot in the middle where the table and culet are opposite. All the light falling on the front of the stone is reflected from the facets, and the light passing into the diamond is reflected from the interior surfaces and refracted into colours when it passes out into the air, giving rise to the lightnings and coruscations for which the diamond is supreme above all other gems.

I hold some of Mr. Streeter's magnificent diamonds in the electric light, and by transmitted light you will see they are black, while by reflected light they fill the room with radiance and colour.

The accompanying table gives the refractive indices of diamonds and other bodies. (See next column).

According to Dr. Gladstone, the specific refractive energy,—

$$\frac{\mu - 1}{d},$$

will be for the D line 0.404, and the refraction equivalent,—

$$P \frac{\mu - 1}{d},$$

will be 4.82.

After exposure for some to the sun many diamonds glow

Refractive Indices for the D Line.

Chromate of lead	2.50—2.97
Diamond	2.47—2.75
Phosphorus	2.22
Sulphur	2.12
Ruby	1.78
Thallium glass	1.75
Iceland spar	1.65
Topaz	1.61
Beryl	1.60
Emerald	1.59
Flint glass	1.58
Quartz	1.55
Canada balsam	1.53
Crown glass	1.53
Fluor-spar	1.44
Ice	1.31

in a dark room. Some diamonds are fluorescent, appearing milky in sunlight. In a vacuum, exposed to a high-tension current of electricity, diamonds phosphoresce of different colours, most South African diamonds shining with a bluish light. Diamonds from other localities emit bright blue, apricot, pale blue, red, yellowish green, orange, and pale green light. The most phosphorescent diamonds are those which are fluorescent in the sun. One beautiful green diamond in my collection, when phosphorescing in a good vacuum, gives almost as much light as a candle and you can easily read by its rays. The light is pale green, tending to white.

(To be continued).

CATHODE RAYS AND SOME ANALOGOUS RAYS.*

By SILVANUS P. THOMPSON, D.Sc., F.R.S.

1. THE size of the cathodic shadow of an object depends upon its own electric state, as already found by Crookes (*Phil. Trans.*, 1879, Part II., p. 648). If it is negatively electrified the shadow expands. If it is positively electrified the shadow contracts. The position, as well as the size of a cathodic shadow, may be affected electrostatically, the rays which cast the shadow being repelled from a neighbouring body if the latter is negatively electrified. In some cases the contraction of the shadow of a narrow object that is made positively electrical (anodic) may go so far that the luminous margins approach and even overlap, giving the appearance of a bright or negative shadow in place of a dark one. The enlargement of a shadow when the object is made cathodic, and the diminution of the shadow when the object is made anodic, both depend upon the degree of exhaustion of the tube, and both are augmented up to a certain point by raising the degree of exhaustion. They are also unequal, the enlargement when the object is made cathodic vastly surpassing the diminution when the object is made anodic, other things remaining equal. The conclusion is reached that cathode rays are capable of being deflected electrostatically, being apparently strongly repelled from a neighbouring cathodic surface, and less strongly attracted towards a neighbouring anode. Incidentally it was observed that two cathode beams from two small disc cathodes can cross through or penetrate one another without interfering with another.

2. The electrostatic deflection of cathode rays by an electrified object is found to be dependent upon the surface of that object as to whether it is a conductor or not. Objects protected by a non-conducting layer of glass do not at moderately low exhaustions, when made cathodic, repel or deflect cathode rays, and their shadow does not

* Abstract of a Paper read before the Royal Society, June 17, 1897.

enlarge. But at a certain minimum exhaustion they suddenly exert an electrostatic deflection. Naked objects made cathodic deflect the cathode rays at all exhaustions.

3. The "splash" phenomenon often observed on the tube-wall of a Crookes tube, where it is struck by the cathode beam, at the stage of exhaustion a little below that which suffices to evoke Röntgen's rays, is due to electrostatic deflections of the cathode rays by the charges on the glass.

4. A hot wire used as an object casts a cathodic shadow precisely like that of the same wire cold. Under some circumstances, if the wire is heated by an electric current, the difference between the electrostatic state of its different parts may slightly affect the size of the shadow it casts.

5. Cathode rays cannot be concentrated by reflection either from a non-conducting or a conducting surface, nor by passage through a metal tube which is itself negatively electrified.

6. When cathode rays strike upon an internal metal target or anticathode there are emitted from the latter (both at exhaustions lower than suffice to produce Röntgen rays, and at exhaustions at which those rays are also produced) some internal rays resembling ordinary cathode rays in the following respects:—They produce a similar luminescence of the glass; they cast shadows of objects; they are susceptible of deflection both magnetically and electrostatically. But they produce no Röntgen rays where they fall upon the glass surface. They do not follow either the law of specular reflection, nor that of diffuse reflection, but are emitted from the anti-cathode surface apparently according to a similarly anomalous distribution to Röntgen's rays, *i.e.*, with nearly equal intensity, at all angles up to 90° with the normal. It is proposed to call these rays para-cathodic rays in contradistinction to the ordinary or ortho-cathodic rays. From the similarity of their distribution with that of the Röntgen rays it is inferred that the physical processes concerned in their production are identical. These para-cathodic rays are emitted from the anti-cathode both when the latter is made an anode and when it is neutral or even made cathodic. From an anti-cathode there may proceed at one and the same time, and in one and the same direction, para-cathodic rays and Röntgen rays, which, meeting an interposed object, may cast simultaneously two shadows—a para-cathodic shadow on the glass, and a Röntgen shadow on an external screen of barium platinocyanide. The former shadow can be deflected by a magnet, the latter cannot. The former shadow expands if the object is made cathodic, the latter does not.

7. If thin metal screens are used to sift the cathode rays, the luminescent phenomena change. The rays of least penetrating power appear to be most susceptible to magnetic and electrostatic forces. The various constituents of a heterogeneous cathode beam are emitted in various proportions at different degrees of exhaustion. In the cathode rays emitted at higher degrees of exhaustion there is a greater proportion of the less deflectable rays. The least deflectable rays are those which most readily penetrate through a perforated screen when that screen is itself made cathodic.

When ordinary cathode rays fall upon a perforated screen which is itself made cathodic, or are attempted to be passed through a tubular cathode, there emerge beyond the screen or tube some rays, here termed dia-cathodic rays, which differ from the ortho-cathodic and also from the para-cathodic rays. These dia-cathodic rays are not themselves directly deflected by a magnet. They show themselves as a pale blue cone or streak. Where they fall on the glass they do not excite the ordinary fluorescence of the glass. The dia-cathodic rays excite, however, a different or second kind of fluorescence, the tint in the case of soda-glass being a dark orange. Intervening objects in the beam or cone of dia-cathodic rays cast shadows. The orange fluorescence evoked on soda-glass by the dia-cathodic rays shows in the spectroscopic

the D lines of sodium only. The shadows cast by dia-cathodic rays are not deflected by the magnet, nor do they change their size when the object is electrified.

ON THE CHANGE OF ABSORPTION PRODUCED BY FLUORESCENCE.*

By JOHN BURKE, B.A. (Dub.),
Berkeley Fellow of the Owens College, Manchester.

If a body, A, of some fluorescent substance, such as uranium glass, be transmitting light from a similar body, B, which is fluorescing, the amount of light transmitted by A from B seems quite different, according as A is fluorescing or not. There appears to exist in a dilute solution of fluorescein or eosine a small difference, but a strong solution of either does not permit its fluorescent light to penetrate, except a very small thickness of the liquid, whether fluorescing or not.

Sulphate of quinine is too transparent to the luminous rays, so that even if a change did exist it could hardly be detected. The case is otherwise with uranium glass, in which the effect is well marked.

The compounds of uranium are remarkable for many characteristic properties connected with fluorescence, which they seem to exhibit more readily than most other bodies. The spectrum of the fluorescent light emitted by uranium glass shows certain maxima and minima, as was noticed by Stokes in 1852.

The experiments which have been carried out with a view to testing whether a change in the absorption is produced by fluorescence, have been almost exclusively upon uranium glass. Regarding it as the most suitable substance to experiment upon, a careful series of experiments have been made to determine whether the phenomenon really existed or not.

If we call α and β the fractions of the light from B transmitted by A, according as the latter is fluorescing or not, for uranium glass 1 c.m. thick; the values of α in a particular set of experiments, in which eye determinations were taken, were as follows. Each experiment consisted of twenty observations.

Exp. I.	..	..	..	$\alpha = 0.54$
II.	..	..	..	$= 0.46$
III.	..	..	..	$= 0.51$
IV.	..	..	..	$= 0.36$

and for β —

Exp. I.	..	..	..	$\beta = 0.70$
II.	..	..	..	$= 0.84$
III.	..	..	..	$= 0.72$
IV.	..	..	..	$= 0.92$

The mean value of α obtained from these being $\alpha = 0.47$, $\beta = 0.79$.

The ratio $\frac{\beta}{1+\alpha}$ was also independently determined, and found as the mean of eighty observations to be 0.507.

The values of α and β have also been determined photographically, giving—

$$\alpha \begin{matrix} > 0.48 \\ > 0.43 \end{matrix} \quad \beta \begin{matrix} < 0.75 \\ > 0.89 \end{matrix}$$

If we take the maximum value of α given photographically 0.48, the ratio $\frac{\alpha}{1+\alpha} = 0.32$, and instead of obtaining equality when the photometer is adjusted for this value the difference is most marked.

The effect has also been shown by obtaining two photographs on the one plate; one photograph being the result of an exposure to the light from two fluorescing cubes one behind the other, and the second photograph the result of superposing the effect of the light from A

* Abstract of a Paper read before the Royal Society, June 17, 1897

alone, when fluorescing, upon that from B after having passed through A, when the latter was not fluorescing. The exposure in each of the three cases being the same, a very distinct difference is shown in the result; the superposed photographs being always the darker in the negative, notwithstanding the fact that the resultant effect of superposing two photographs due to light of the same intensity, or nearly so, has been found not to be equal to, but less than, that due to light of double the intensity acting for half the time. If the resultant effect were equal to the sum of the separate ones, the effect caused by the change of absorption would have been still more marked.

In the determinations of α and β a null method has been employed, by which any appreciable want of uniformity in the illumination can be detected.

The source of illumination has been almost invariably the spark discharge of a Leyden jar between cadmium electrodes, being one of the richest sources of the fluorescence exciting rays, and the photometer one specially constructed for the purpose.

ON THE VOLUMETRIC DETERMINATION OF ZINC BY POTASSIUM FERROCYANIDE.*

By L. L. DE KONINCK and EUG. PROST.

I. Introduction.

THE volumetric estimation of zinc has, since the publication in 1856 (*Polytechnisches Journal de Dingler*, vol. cxi., p. 114, and vol. cxliii., p. 263; *Journal de Pharmacie*, [3], vol. xxix., p. 205, and vol. xxxi., p. 70), by Max Schaffner, of the process which now bears his name, been the subject of numerous researches, which have had for object either the improvement of this process—which at first left much to be desired—or the discovery of others.

This fact is easily accounted for by the importance of having, especially in the laboratories at mines and zinc works, a trustworthy commercial method, uniting rapidity and simplicity with a sufficient degree of exactness with regard to the value of the metal.

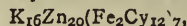
Of all the methods proposed, two only appear to be now in general use.

On the Continent, so far as we know, the Schaffner process is almost exclusively used; it is even the only volumetric one described, in several English and French books, among the recent methods for the analysis of zinc ores (Riche and Gelis, Silva, Halphen; Clowes and Coleman). In Germany it is certainly the most widely-used process. In America, however, it is otherwise, for, according to an interesting report made to the Scientific Society of Colorado (*CHEM. NEWS*, vol. lxvii., pp. 5 and 17, 1893) by a Committee charged specially to endeavour to establish uniformity among the methods used in that country, it would appear to be the Galletti process (*Bull. Soc. Chim.*, vol. ii., p. 83, 1864) modified by Fahlberg (*Zeitsch. f. Anal. Chem.*, vol. xiii., p. 379); that is to say, the process based on the precipitation of the zinc by ferrocyanide of potassium, in an acid solution, which is generally preferred in the mining districts; it is, in fact, the only one cited by the chemists who were consulted. It is also recommended by G. E. Dougherty, in a note on the Analysis of Minerals (*Engineering and Mining Journal*, 1890, p. 178; *Zeitsch. f. Angew. Ch.*, vol. iii., p. 306, 1890).

The methods of Schaffner and Galletti are the only ones which are found in the best-known treatises on

quantitative analysis of late years (Kerl, Balling, Post, Böckmann, &c.).

The sulphide of sodium process of Schaffner has been carefully studied, and thoroughly described in detail. Most writers are in accord on the details of manipulation, and notably on the use of *Polka* paper (salts of lead) as an indicator. This unanimity does not exist in the case of the ferrocyanide method. In this case the different writers are not even agreed as to the formula of the reaction on which the method is based. According to some the precipitate obtained should be simple ferrocyanide of zinc, $Zn_4Fe_2Cy_{12}$; according to others it should be the double ferrocyanide of zinc and potassium, $K_2Zn_3Fe_2Cy_{12}$, described long ago by Mosander (*Handwörterb. d. rein. u. Angew. Ch.*, 1848, p. 86). Others, again, maintain that the exact composition of the precipitate is more complicated still, and corresponds to the formula—



or should be even more or less indeterminate (Zulkowski, *Polyt. Journ. de Dingler*, vol. ccxlix., p. 175, 1883), for in spite of the high state of purity in which it is possible to obtain ferrocyanide of potassium, and the almost absolute stability of this reagent, they recommend the experimental determination of the relations between this salt and the quantity of zinc precipitated, this proportion not being, according to them, so simple as is generally expressed by the formulæ in common use (Mohr, Classen, 1886, p. 459).

A priori, according to the description of the process given by most writers, this would seem to be of extremely simple application, while such is not absolutely the case with the Schaffner process. Why, therefore, is the latter preferred in our continental laboratories? Is it merited, or simply a matter of custom?

It is this question that we have set ourselves to solve: one of us having, owing to a previous appointment, had a large experience in Schaffner's method, we were enabled to speak with authority when we had compared the results obtained from the careful study we made of the ferrocyanide process.

It has been proposed to apply, to the volumetric estimation of zinc, precipitation under the three following conditions:—

1. In acid solution (Galletti);
2. In simple ammoniacal solution (A. Renard);
3. In a tartaric-ammoniacal solution (Giudice).

From these spring three different processes, which we will designate by the names of their respective authors, and which we hope to study successively. We take first the Galletti process, the oldest and most widely used of the three.

II. Historical.

It was in 1864 that M. Galletti described for the first time (*Mémoire* presented to the Royal Academy of Science of Turin, 1864—*Bull. Soc. Chim.*, vol. ii., p. 83, 1864) the method of estimating zinc by means of ferrocyanide, a process which he had, however, foreshadowed some years previously, on the occasion of publishing an analogous process for the estimation of copper (*Ibid.*, 1856). After first separating the iron by ammonia, he added acetic acid to the filtrate, heated to 40°, then ran in the titrated solution of ferrocyanide; the end of the reaction was indicated by the sudden milky appearance of the liquid. The titration recommended is $T_{12} = 0.01$ grm. Galletti admits that the precipitate is the simple zincic ferrocyanide, and he attributes to impurities in the reagent used the fact that the results do not agree with theory, in spite of the very wide differences found. He warns one against acidulating with mineral acids, any excess interfering with the reaction, which, he says, can be perceived by the yellow colour of the liquid, due to ferrocyanide which has not been acted on. This colour does occur, but Galletti is mistaken as to its origin: we shall show later on to what this is due, and how it can be avoided. In another note published in 1868 (also *Zeitsch.*

* In the *CHEMICAL NEWS* (vol. lxxv., p. 182) we published a short abstract of this paper; but we regret to say that the copy which came into our hands was incomplete and inaccurate. We therefore think it best to now insert the paper in full, or at any rate but slightly abridged.—*Ed. C. N.*

f. Anal. Chem., vol. viii., p. 135, 1869) he returns to the application of his process to the assay of minerals, with a view to modifying somewhat the method of preparing the zinc solution; but the actual estimation of zinc is done in exactly the same manner. The presence of lead has no influence; manganese is eliminated by adding bromine to the ammoniacal solution, and exposing it to the air for twenty-four hours. We shall soon come to the influence this reagent exerts. Finally, the operation can be carried on without filtering off the iron.

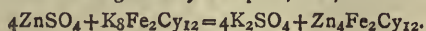
In 1874 C. Fahlberg published, as new (*Zeitsch. f. Anal. Chem.*, vol. xiii., p. 379, 1874), without referring to Galletti, the ferrocyanide process; his method of procedure was different to that of his forerunner, in that the precipitation was effected in hydrochloric solution, and, above all, by the means employed for detecting the end of the reaction. Fahlberg here utilises, by inverting it, the method used in the titration of phosphates by a titrated solution of a uranium salt,—that is to say, he uses the spot method, of a solution of uranic nitrate, to detect the brown colour which is produced the moment the potassic ferrocyanide commences to predominate. According to him, and here he is wrong, the presence of manganese would be without influence if the solution is sufficiently acid, so he does not eliminate it before titration. In each experiment the solution was acidulated with 10 to 15 c.c. of hydrochloric acid (sp. gr. 1.12 = 23.82 per cent), or 2.7 to 4 grms.; this is too much. He recommends, as does Galletti, the preparation of a solution of ferrocyanide, T = 0.01 gm., and the experimental determination of its strength by titrating with a solution of pure zinc dissolved in HCl; to this solution he also adds 5 parts of ammonium chloride for each 1 part of zinc, so as to obtain a precipitate "as attenuated as possible," which will subside rapidly without "carrying down potassium ferrocyanide." Fahlberg always describes the precipitate as a zincic ferrocyanide; he seems to believe, from what we have just quoted, that such is really the case.

To estimate zinc in an ore, he dissolves it in aqua regia, with excess of HCl, precipitates the copper, &c., by sulphuretted hydrogen, re-oxidises the iron salts with nitric acid, precipitates with excess of ammonia, and filters. The ammoniacal filtrate is made acid with HCl in the proportion mentioned above; the titrated solution of ferrocyanide is then run in, little by little, until the nitrate of uranium test shows the end of the operation.

Thus, as we shall show directly, the method of working described above contains sources of error which the author has quite overlooked; so it is not astonishing that the results published by him—results obtained from eleven ores from the Hartz district, containing from 3 to 23 per cent of zinc—should not be of great value; compared with those obtained dosimetrically they show errors reaching 0.49 per cent at least, on 19.77 per cent, and as much as 0.33 per cent on 3.57 per cent. This is very far from the exactness we have a right to expect, and that which can be obtained by Schaffner's method.

In the same year, 1874, Galletti published a third edition of his pamphlet (Gênes, 1874); the only modification indicated refers to the proportion of ammonia and acetic acid, to be used in a case when one wishes to titrate the zinc without previously removing the precipitated iron.

When using a zincic solution with alkaline ferrocyanides K. Zalkowski, like Fahlberg, seems to have entirely overlooked the work already done by others. When using a normal solution of ferrocyanide (105.5 grms. per litre), and a deminormal solution of a pure zincic salt, such as zinco-potassic sulphate, $K_2SO_4 \cdot ZnSO_4 \cdot 6H_2O$ (mol. weight 442.57, 110.82 gm. per litre), this author readily recognises the fact that the reaction between the zinc salt and the ferrocyanide is not so simple as would appear from the formula which is generally accepted, viz.,—



(To be continued).

METHOD OF ESTIMATING ALDEHYD IN ETHER.

By M. FRANCOIS.

THE almost constant presence of ordinary aldehyd in so-called pure ethers, even that used for anæsthetic purposes, does not yet appear to have attracted sufficient attention from pharmacologists. This impurity, however, exists in a sufficiently high proportion in the products ordinarily delivered to pharmacists, and ether containing several units per cent of aldehyd is not unfrequently met with.

Thus, in medicine we admit a high proportion of impurity, which would not be tolerated in any alcoholic industry. M. Adrian (*Moniteur Scient.*, 1894, p. 835) in an excellent note, which completed the well known work of Regnault and Adrian on the estimation of ether, noted this constant presence of aldehyd, and gave a method for detecting and eliminating it. This method consisted of passing a current of dry ammonia gas through the cooled ether; crystals of aldehyde of ammonia were deposited, insoluble in ether. By this means we can detect the presence of 0.5 per cent of aldehyd. The method of purification consists of filtering and then eliminating the ammonia and rectifying.

It occurred to me to make use of the action of bisulphited rosaniline for the estimation of aldehyd in ether, and to apply to this estimation the colorimetric method of Mohler, who has rendered such great services in the analysis of alcohols. In this method (Mohler, *Moniteur Scient.*, 1890, p. 893, and 1891, p. 582) we make use of the reddish violet colouration produced by the action of bisulphited rosaniline on aldehyd. We cause this reagent to act on an alcohol, whose contents of aldehyd is known, and also on the alcohol under examination; the tints developed are observed with a Duboscq colorimeter, and by dilution we can bring the two alcohols to the same colour. From the amount of dilution we can calculate the weight of aldehyd per litre present.

To apply this method to the estimation of an ether, it is necessary to make some modifications. Mohler's reagent, which contains bisulphite of soda, precipitates sodic salts with strong alcohol and with ether; further, it will not mix with ether. We can get over this difficulty by using, for the preparation of the reagent, an aqueous solution of sulphurous acid instead of bisulphite of soda, and by adding to the ether, at the moment of making the test, its own volume of 95 per cent alcohol, free from aldehyd. With these modifications, the mixture of ether, alcohol, and reagent remains limpid, and the colorimetric determinations are easily made.

Preparation of the Reagent.—A reagent of medium sensitiveness can be obtained by using:—

Water recently saturated with SO ₂	..	..	220 c.c.
Solution of fuchsine, 1/1000th	..	..	30 "
Sulphuric acid, 66°	..	..	3 "

The solutions of fuchsine and sulphurous acid are first mixed, and after agitation we add the sulphuric acid.

The reagent should be colourless. If necessary, we can filter after twenty-four hours. The fuchsine used should be the ordinary kind, not acid (Cazeneuve, *Journ. de Pharm.*, June 15, 1896). The reagent is the more sensitive as it contains less sulphuric acid. Its sensitiveness can thus become exaggerated, even to the extent of becoming coloured when mixed with absolutely pure alcohol and ether. Increasing the quantity of sulphuric acid causes this fault to disappear.

A mixture of 5 c.c. of pure ether, 5 c.c. of pure 95 per cent alcohol, and 4 c.c. of the reagent remained uncoloured for fifteen minutes. If we replace the pure ether by an ether containing more than 1/10,000th part of aldehyd, the mixture assumes a red-violet colour, the more intense as it contains more aldehyd, but not in direct proportion.

To perform the experiment it is necessary to have some freshly-made reagent, ether free from aldehyd,

solution of aldehyd in 90 per cent alcohol containing 1 grm. of aldehyd per litre, a similar solution containing 0.1 grm. of aldehyd per litre, and a Dubosq colorimeter.

Alcohol containing 1/1000th of aldehyd will serve as a comparison for all ethers containing more than 1/1000th of aldehyd, and alcohol at 1/10,000th for ethers containing from 1/1000th to 1/10,000th of aldehyd.

Method of Estimation.—Into a test-tube (1) we pour 5 c.c. of alcohol containing 1/1000th part of aldehyd and 5 c.c. of pure ether; into a second tube (2) we pour 5 c.c. of alcohol containing 1/10,000th of aldehyd and 5 c.c. of pure ether; into a third tube (3) we pour 5 c.c. of pure 95 per cent alcohol and 5 c.c. of the ether to be examined. At the same moment we add to each 4 c.c. of the reagent, shake well, cork, and note the time. After fifteen minutes observe the colour produced.

We note in the first place if the intensity of the colouration is the stronger in tube 3 than in tube 1; if it be stronger, we know that the ether contains more than 1/1000th of aldehyd, and we therefore take tube 2 as the standard of comparison; if the colour be less strong, we know that the ether contains less than 1/1000th of aldehyd, and we take tube 2 as the standard.*

In the case of an ether containing more than 1/1000th of aldehyd we note the colour through a thickness of 10 m.m.

If N be the thickness of the solution under examination (tube 3), which has the same intensity of colour as the standard of comparison, the proportion x per litre of aldehyd, taking the amount present as proportional to the colour, will be given by the equation—

$$\frac{x}{1 \text{ grm.}} = \frac{10 \text{ m.m.}}{N}$$

The proportion 10/N indicates how much the ether under examination should be diluted with pure ether, so that its contents in aldehyd should be brought to 1 grm. per litre.

We now re-commence the estimation, taking tube 1 as the highest, and preparing tube 3 with ether thus diluted, and the value of N will be very near to 10 m.m. The new proportion 10/N enables us to make another dilution, more exact, and then to make a third and last trial which will give a complete uniformity of tints. From the quantity of pure ether it has been necessary to add, we deduce the quantity of aldehyd present in the ether under examination.

For an ether containing from 1/10,000th to 1/1000th of aldehyd, we operate in the same manner, but taking tube 2 as the standard; that is to say, a solution of aldehyd of 1/10,000, and comparing the colours through a thickness of 25 m.m.

This method enables one to estimate very small quantities of aldehyd, and it can be done in less than an hour.

Preparation of Ether free from Aldehyd.—This is easily prepared by the action of permanganate of potash in alkaline solution on ether at 65°. We use anhydrous commercial ether for choice. This is placed in a stoppered litre flask with 200 c.c. of a saturated solution of permanganate of potash and 20 grms. of caustic soda. After twenty-four hours and frequent agitation, we decant by means of a funnel fitted with a tap, and submit the ether to the same treatment over again. The filtered ether is then left for twenty-four hours in contact with a mixture of 50 grms. of quicklime and 50 grms. of fused chloride of lime; it is then filtered and distilled. The permanganate has oxidised the ether and the alcohol, and the ether thus obtained gives no colouration with the aldehyd reagent, at least not after fifteen minutes.

Preparation of Alcohol free from Aldehyd.—Commercial alcohol is freed from aldehyd, furfural, and bases by adding to 1 litre of the alcohol 10 c.c. of aniline and 10

c.c. of phosphoric acid at 45° B., boiling for an hour (with a vertical condenser), and distilling.—*Journal de Pharmacie et de Chimie*, Series 6, vol. v., No. 11.

THE SMALL BESSEMER PROCESS FOR STEEL CASTINGS.

By SERGIUS KERN, M.E., St. Petersburg.

In the *CHEMICAL NEWS*, vol. lxxiii., pp. 170, 192, we published some notes on the Walrand-Légénisél small Bessemer process. At the Baltic Shipbuilding Works, St. Petersburg, for some time past, we have used instead of the Walrand process the usual Bessemer method for making steel castings in our convertors, with a capacity of two-thirds of a ton each.

The chief reasons which induced us to discontinue the use of the *modus operandi* advised by the Walrand process are as follows:—

1. The uncertainty of obtaining uniform results. Steel castings obtained by the Walrand process may be good for the general trade, but may be variable when tested for tensile strength and elongation.
2. The extreme difficulty in obtaining steel castings which would give regular results, after being mechanically tested according to the regulations of the Russian Naval Technical Committee (28 to 35 tons per square inch of tensile strength, 14 per cent of elongation in 2 inches).
3. No necessity for the addition of ferro-silicium during the blow, in order to raise the temperature of the metal in the convertor, before the casting. In our convertors, as mentioned above, we produce, running the process by the usual Bessemer method, steel at a temperature of 2200°—2400° C., quite sufficient for our shipbuilding castings, weighing one cwt. and higher.
4. Starting with a metal from the cupola containing 4 per cent of silicon, we only lengthen the process, and make also the product obtained dearer. We start the process now, with a metal from the cupola, with 2.25 per cent of silicon, which we find quite sufficient for the charge of our convertors.
5. We certainly do not use from the very commencement, as I pointed out, Mr. Walrand's system to put the blast on, and turn the convertor up for an instant, after making the final additions, in order to mix them with the metal. Such a serious mistake will give metal with blowholes in very soft steel, or, in other cases, secondary reactions.

The following are some details of our present work while making steel castings for Government orders;—

Charges Nos. 63 and 64. (Nearly equal operations).

Charge in cupola:—

Pig-iron, Ayresome, with 4% silicon .. 900 lbs.
Runners 700 „

(This charge was melted in the cupola in 40 minutes, and run into the vessel).

Minutes from the commencement of blow.

9	D line visible; pressure 7½ lbs.
10	Green lines visible; pressure from this point to end of the operation 5½—5 lbs. per sq. inch.
18	No green lines visible; convertor turned down.

Three minutes after the appearance of the D line the convertor was turned down and the slag cleaned off. This operation took 2 minutes; time not counted in the above table.

After the disappearance of the green lines, 12 pounds of ferromanganese, containing 80 per cent of manganese, were added to the convertor, after cleaning the slags off a second time. A further charge of aluminium was made, and the steel, after the usual tests, was run into the

* It would seem to be more regular to take as standards of comparison solutions of aldehyd in pure ether; but we used solutions of pure alcohol because they last longer without changing. The results are evidently the same.

casting ladle, into which aluminium was also thrown. In all 2½ pounds of this metal was used.

The metal had a temperature of 2400° C. Prof. Wiborgh's very handy and reliable thermophone combs were used. The metal cast very quietly, and did not rise in the gates of the moulds. A casting weighing 8 cwts. was made out of the charge No. 63, and from No. 64 other castings of different weight were made.

Out of a charge of 1600 pounds in the cupola about 1200 pounds is obtained as finished steel.

The following are the chemical and mechanical analyses of our steel castings:—

	Carbon	0.11 per cent	
	Manganese	0.45 "	
	Silicon	0.08 "	
No. of specimen.	Tensile strength: Tons. per sq. in.	Elongation in 2 ins. P.c.	Bending test. Angle.
No. 63, annealed..	25.1	20.8	141 (a)
Ditto	26.2	15.8	
No. 64, annealed..	23.7	31.6	50 (b)
Ditto	23.5	30.1	
No. 64, unannealed	23.0	22.6	40 (c)
No. 64, forged specimens	30.3	27.8	124 (d)
Ditto	30.1	27.8	

Remarks.

(a) Bent without cracks. (b) Cracked. (c) Broke with a coarse crystalline fracture, one edge coloured brown. (d) Without cracks, there being one crack coming in forging.

In conclusion, we must state that the working out of many details and improvements gives full credit to the ingenuity of Mr. Leo Reyher, the manager of the Steel Foundry of the above-mentioned Russian Government Works.

June 14, 1897.

HONOURS FOR MEN OF SCIENCE.

THE Honours list issued on Tuesday, June 22nd, in connection with the Diamond Jubilee, contains the names of a number of men of science upon whom Her Majesty has been pleased to confer distinctions.

Dealing first with Fellows of the Royal Society, Mr. Crookes and Dr. Gowers receive knighthoods. In the Order of the Bath, Mr. Wolfe Barry, President of the Institution of Civil Engineers, Dr. Frankland, Foreign Secretary of the Royal Society, Dr. Huggins, Mr. Norman Lockyer, Director of the Solar Physics observatory, Dr. Thorne Thorne, Principal Medical Officer to the Local Government Board, and (naval promotion) Admiral Wharton, Hydrographer of the Admiralty, are appointed K.C.B.

Mr. Christie, Astronomer Royal, and Mr. Niven, Director of Studies at the Royal Naval College, are appointed C.B.

In the Order of the Star of India, Sir Joseph Hooker and Lieut.-General Strachey are promoted to the grade of G.C.S.I.

In addition to the foregoing, Baronetcies are conferred upon Sir Wm. MacCormac, President of the Royal College of Surgeons; Mr. Wilks, President of the Royal College of Physicians; and Mr. Thomas Smith, Surgeon-Extraordinary to Her Majesty. Mr. Durston, Engineer-in-Chief to the Navy, is made a K.C.B., and knighthoods are conferred upon Mr. A. R. Binnie, the Engineer to the London County Council, and Dr. Felix Semon.—*Nature*, June 24, 1897, p. 181.

Preparation of Furfurane.—P. Freundler.—Furfurane may be obtained pure and in a good proportion by heating pyromucic acid in a closed vessel to 260°—275° for two hours.—*Comptes Rendus*, cxxiv., No. 21.

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, June 25th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

A PAPER by Mr. SUTHERLAND, on "*A New Theory of the Earth's Magnetism*," was taken as read.

Dr. KUENEN described some "*Experiments on Critical Phenomena*," made in continuation of a research on the condensation and critical phenomena of mixtures of ethane and nitrous oxide, the results of which were published last year. The author now investigates mixtures of ethane and acetylene, and mixtures of ethane and carbonic acid, and finds for them similar properties to those of the mixtures of ethane and nitrous oxide. The first part of the paper refers to the preparation of ethane, and the effect of impurities on its vapour pressure and critical constants. Ethane from ethyl iodide is not very pure; it has generally several per cent of an admixture of a substance of higher critical temperature and higher density than ethane: this substance is probably butane. Ethane from sodic acetate, by electrolysis, is nearly pure; the method of preparation is described by the author. The pressure and corresponding volumes during condensation are given for this substance at various temperatures. In the former paper, above referred to, instances are mentioned of mixtures having critical temperatures *below* those of the component substances. The only instance of critical temperatures *above* those of the components seem to be those relating to mixtures of carbonic acid and acetylene. According to an experiment of Dewar's, however, a mixture of $\frac{1}{2}$ CO₂ and $\frac{1}{2}$ C₂H₂ has a critical temperature of 41° C., those for carbonic acid and acetylene being 31° and 37° respectively. The present investigation contradicts this result. Dewar may not have taken sufficient precaution in avoiding errors of retardation. Mixtures of carbonic acid and acetylene have critical temperatures between those of the component pure gases. The diagram connecting temperature and volume shows that the plait-point curve is a line with small curvature; the border-curve is relatively narrow. An instance of a critical temperature above those of the components, for this mixture, has not yet been proved. Theory indicates that this phenomenon probably occurs for mixtures having a minimum vapour-pressure at low temperatures. Critical temperatures below those of components, seem to occur for mixtures having a maximum vapour-pressure, as for nitrous oxide and ethane. The law connecting the two phenomena is deduced from van der Waal's theory. As a further application of this theory, it is shown that in consequence of certain coincidences between the real border-curve and the hypothetical border-curve, the critical point of the maximum mixture may be determined in exactly the same way as for a single substance. A remark is added with regard to the condensation of such substances as exhibit changes of molecular systems. If an association takes place of molecules to more complicated systems, van der Waal's formula does not apply.

Dr. S. P. THOMPSON asked whether diagrams characteristic of cyanogen had been obtained. Its remarkable polymerism suggested an interesting case for critical phenomena.

Dr. KUENEN thought such a substance might be worth investigating.

A paper by Dr. BARTON, on "*The Attenuation of Electric Waves in Wires*," was taken as read.

Mr. G. F. C. SEARLE read a paper on "*The Steady Motion of an Electrified Ellipsoid*."

The first part of the investigation is printed in the *Phil. Trans. Roy. Soc.* It contains the principles required in the solution of problems with respect to moving electrical

charges. The second part, now presented to the Physical Society, deals with the motion of a charged ellipsoid; the treatment is entirely mathematical. When any system of electric charges moves with uniform velocity through the æther, the electro-magnetic field, referred to axes moving forward with the charges, can be completely defined by means of a quantity of which the electric force and the magnetic force are simple functions. Another vector concerned in the problem is the mechanical force experienced by a unit charge moving with the rest of the system. A distribution of electricity on the surface of a charged body, such as to give zero distribution at all points inside the surface, is an equilibrium distribution. Since the mechanical force vanishes inside the surface, it is shown that on the outside of the surface the mechanical force is perpendicular to the surface, and the above-mentioned function is constant over the surface, and the distribution on an ellipsoid is the same for motion as for rest. When a charged sphere is at rest it produces the same effect as a point-charge at its centre. If the sphere is in motion, it produces the same effect as an uniformly-charged line whose length bears to the diameter of the sphere the same ratio that the velocity of the sphere bears to the velocity of light. When the sphere moves with the velocity of light, the line becomes the diameter of the sphere; the same is true for an ellipsoid. At the velocity of light the charge on any surface is in equilibrium, whatever the distribution. The force between two charges moving with the speed of light is zero. The lines of electric force for a charged sphere in motion are not radial; they form a series of hyperbolas. The author proceeds to calculate the total energy possessed by an ellipsoid when in motion along its axis of figure. Expressions are given (1) for the energy of a Heaviside ellipsoid; (2) for a sphere; and (3) for a very slender ellipsoid. In all cases the energy becomes infinite when the charges move at the velocity of light. It would seem impossible to make a charged body move at a greater speed than that of light.

Prof. PERRY said the paper would help to solve many problems connected with the effect of the rotation of the earth upon electrical surface changes. An expression might be found for the mechanical and magnetic forces due to the motion of a charge at any point of the earth's surface. At the equator a point moves at different velocity at midday to its midnight velocity; it might now be possible to determine the magnetic and mechanical effects due to electric charges at equatorial points.

Mr. BLAKESLEY asked whether, in calculating the mutual action of two charged particles, proceeding at the velocity of light, it was assumed that the lines of motion were parallel.

Mr. SEARLE said he had always considered parallel lines of motion; he could not say whether the force would be zero in any other case. The results arrived at in the paper could be applied to problems connected with distributions of terrestrial charges.

The PRESIDENT proposed votes of thanks to the authors; the meeting then adjourned until November.

OBITUARY.

PROF. SCHÜTZENBERGER.

CHEMICAL Science, not merely in France but throughout the world, has sustained a heavy blow by the death of Prof. Schützenberger, at the age of 67. The deceased was a native of Strasburg, where he studied medicine, and became connected with the chemical laboratory of the Conservatoire des Arts et Metiers, of that city. He became successively Assistant-Director of the Sorbonne Laboratory and Head of the Chemical Department of the College de France, where he filled the chemical chair since 1876. Along with these posts, which he held with

success and distinction, he was elected Head of the Paris Municipal School of Chemistry and Physics. In 1884 he became a Fellow of the Academy of Medicine. Lastly, in 1888, on the death of Débray, he was elected a Fellow of the Academy of Sciences.

These honours, and the widely-felt appreciation of which they were the embodiment, were earned by a series of sterling researches in organic chemistry, especially in its application to colours and their uses in the tinctorial arts, in which department he was recognised as one of the best authorities. His works include researches on the physiology of digestion and fermentation, and, above all, a cyclopædia—as we may call it—of tinctorial chemistry, considered both theoretically and practically, and an investigation into the alkaloids.

The late professor was universally respected by all who had the pleasure of his acquaintance. Prof. Schützenberger died on Monday, June 28th.

NOTICES OF BOOKS.

Fresenius's Quantitative Analysis. Vol. ii. Translated by C. E. GROVES, F.R.S. Part IV.

THE *magnum opus* of the late "Grand Master" (Professor Fresenius) is gradually making its appearance in an English guise.

The present issue treats in succession of zinc-dust, of manganese compounds, nickel compounds, iron compounds, uranium, silver, mercury, and lead compounds.

The translator appears to be doing justice to the original. At the same time we cannot approve of the system of publishing the translation of a book in detached portions.

The Study of Technical Chemistry at the Universities and Technical High Schools of Germany. ("Das Studium der Technischen Chemie an den Universitäten und Technischer Hoch-Schulen, Deutschlands"). *Opinions on the German System of Training Chemists*. By Prof. Dr. FERD. FISCHER.

THIS pamphlet contains the opinions of eminent authorities as to the cause of the relative decline of certain English industries. As a whole, they agree that our failure—for as such we must accept it—is due to our imperfect system of higher education. Chemical technology is for the universities the connecting link between science and industry.

Among the heads of German universities there prevails a unanimous opinion that Germany can maintain its industrial work solely in virtue of its chemical eminence. Slight alterations in the curriculum are proposed in some quarters. But the general tendency is rather to intensify than to lighten the course of study.

In the Presidential Address of the Society of Chemical Industry (1895), it was admitted that business ability and capital cannot suffice to uphold the chemical industry of the country without a fundamental scientific technical training.

We have met with chemical manufacturers who know little of chemical science, and do not seek to know more. Some of these employ an analyst who has to limit himself to routine work. To enquire into the cause of any disappointment is a "waste of time."

Chilian Hygienic Review. ("Revista Chilena de Higiene," publicada el Instituto de Higiene de Santiago). Parts II. and III.

THE most important matter in these issues refers to the observance of quarantine, which, in the whilome Spanish republics, is still maintained. In Chili there are two

grades of quarantine. The quarantine of observation is imposed upon every traveller upon entering Chilean territories, which lasts forty-eight hours. During this period the traveller and his belongings are submitted to systematic disinfection according to the rules laid down.

At the moment when the quarantine expires the traveller will receive, from the head of the sanitary department, a certificate which will serve him as a passport. The head of the sanitary department will communicate with the governor of the department to which the traveller is proceeding.

Rigid Quarantine.—If, during the quarantine of observation, there occur suspicious symptoms, the quarantine is made strict. Rigid quarantine is then continued for eight days. If the patient dies, his body, after disinfection, is buried in ground the drainage of which does not enter the stream. If no such ground is obtained, the body will be incinerated. His possessions will be destroyed by fire, with the exception of articles which the head of the sanitary department may designate.

The disinfection of persons will consist in a general soap-bath or in general lotions, with a solution of sublimate at 1/5000.

Dead bodies will receive an injection of solution of sublimate at 1/500, in the stomach, the bowels, and the carotid and femoral arteries, and the body will further be wrapped in a shroud saturated with the same solution.

CORRESPONDENCE.

ESTIMATION OF CARBON IN FERRO-CHROME.

To the Editor of the Chemical News.

SIR,—Professor Arnold's letter is a surprising continuation of what I hoped might prove a helpful discussion. I ought first to disabuse your readers' mind of the personal illusion by which the Professor would create a false relation between us. He says that "until quite recently Mr. Leffler was a student of mine." In the ordinary sense this is not true. It is true that I was a student at the Technical School for three years, but it is nine years this summer since I left; that was some time before Prof. Arnold came to the school. This he can readily verify by appealing to his Senior Demonstrator. In the autumn of 1894 I desired to acquaint myself with the microscopic analysis of steel, and worked under Prof. Arnold two nights a week from September to Christmas, and ceased work at his suggestion on account of the inadequate accommodation. This fact is a very slender thing on which to found the above assertion, and I feel that it in no way obliges me to submit to him any opinion I may form on a subject like the one under discussion.

I pointed out in my last letter that I had never made the bald assertion that his method only gave half the carbon present. The furnace at our disposal certainly only gave about 50 per cent of the carbon, but I stated that on raising the temperature with the blowpipe the remaining 50 per cent was obtained. Plainly, then, our furnace was not hot enough, though it was as hot as combustion furnaces frequently are. The need of the blowpipe was evidence that the temperature was too low, and I have never attempted to deny it.

Prof. Arnold invited a reply to his first letter, by asking the type of furnace and the diameter of the gas-pipe. To this information I attached a few questions and other details, which the Professor has no inclination to follow, although I refer only to certain pages of his own book, "Steel Works Analysis."

Prof. Arnold refers to Mr. Saniter's letter as proof that I have put up straw men. I cannot imagine to what part he refers, but thank him for the reference nevertheless, because it draws my attention to the unprejudiced support Mr. Saniter gives to my assertion. Saniter's third para-

graph reads "I also found that with copper oxide only about half the carbon is obtained." Now I admit that a little higher temperature is needed for combustion with copper oxide than with lead chromate; but copper oxide will give perfectly accurate results, and failed to do so in Mr. Saniter's hands only because his furnace was not hot enough, which supports my assertion that our furnace is as hot as those generally used, but not enough to completely eliminate the carbon from a mixture of lead chromate and ferro-chrome.

The statement made was not inaccurate, nor was it hastily arrived at, as the method was given a long and fair trial, and the results obtained accurately and honestly recorded.—I am, &c.,

R. L. LEFFLER.

The Laboratory,
Messrs. Thos. Firth and Sons, Lim.,
Sheffield, June 21, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 22, May 31, 1897.

Liquefaction of Fluorine.—H. Moissan and J. Dewar.—This memoir has been inserted in full.

Part Played by Humic Matter in the Fertilisation of Soils.—Armand Gautier.—The algae as well as the microbia which solidify nitrogen find in the humus of the soil and generally in the organic matter of excrements a nutrient which allows of their rapid development. I do not assert that soils owe their fertility to the direct absorption by plants of organic matter, ternary or quaternary.

Purification of Cerium.—M. Wyruboff and A. Verneuil.—Already inserted.

Remarks on the above Communication by Wyruboff and Verneuil.—Already inserted.

Alloys of the Silver-copper Group.—F. Osmond.—The conception of Matthiesen, who saw on certain alloys solidified solutions of allotropic form, whilst in need of certain restrictions, seems to remain vital and fruitful. I do not see that it is opposed to the researches of H. le Chatelier, whose conclusions would receive from it a character of greater generality.

Phosphorescence of Strontium Sulphide.—José Rodriguez Mourello.—We see that an oxidising principle is requisite, as well as a peculiar structure, for strontium sulphide to be susceptible of phosphorescence without omitting the substances whose influence on the property in question is direct or positive.

Contribution to the Study of the Preparation of Common Ether.—L. Prunier.—In the ordinary preparation of ether it escapes, in virtue of its great volatility, in the midst of a heterogeneous medium, unstable, and in perpetual transformation.

Certain Compounds of Phenylhydrazine with Metallic Chlorides.—J. Ville and J. Moitessier.—Phenylhydrazine combines with different metallic chlorides, yielding compounds containing 1 mol. of chlorides with 2 mols. of phenylhydrazine.

Apparatus for the Industrial Analysis of Gases.—Leo Vignon.—This memoir requires the accompanying illustration.

Decomposition-products of Calcium Carbide, and on its Use as an Insecticide.—E. Chuard.—The author has obtained a phospho-carbide possessing powerful insecticide properties.

No. 23, June 8, 1897.

Action of Light on Mixtures of Chlorine and Hydrogen.—Armand Gautier and H. Hélier.—This paper will be inserted at some length.

Observations on the Limitation of Chemical Reactions, with reference to Armand Gautier's Communication.—This memoir will also be inserted in *extenso*.

Memoir by M. Berthelot accompanying the Presentation of his work on Thermo-chemistry.—A kind of preface to the author's recent works.

Examination of certain Spectra.—Lecoq de Boisbaudran.—A controversial paper, directed against Drs. Eder and Valenta. Except the small ray 563·8, seen with K_2SO_4 , but not with K_2CO_3 , all the rays of the author's diagram are seen with K_2SO_4 and K_2CO_3 , and have not been observed with the salts of Na.

NOTES AND QUERIES.

*. * Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Annual Consumption of Chemicals.—I should be extremely obliged if any reader would kindly let me know what is the average amount of sulphate of barium and sulphurite of sodium consumed annually in this country, and in what industries these products are chiefly used.—L. ALSATINI.

TO CORRESPONDENTS.

Eugene Ackermann.—Please forward address; a letter awaits you.

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THOMAS W. SHORE, Warden.

PATENTS, DESIGNS, AND TRADE MARKS ACTS, 1883 TO 1888.

NOTICE IS HEREBY GIVEN, that HIPPOLYTE EUGENE SERULLAS, of 20, Rue Molieres, Paris, France, has applied for leave to amend the Specification of the Letters Patent No. 11166 of 1892 for "Improvements in or connected with an Apparatus for the Obtainment or Extraction of Gutta-percha or the like."

Particulars of the proposed amendments were set forth in the Illustrated Official Journal (Patents), issued on the 16th June, 1897.

Any person, or persons, may give notice of opposition to the amendment (on Form G) at the Patent Office, 25, Southampton Buildings, London, W.C., within one calendar month from the date of the said Journal.

(Signed)

C. N. DALTON,
Comptroller General.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1963.

DIAMONDS.*

By WILLIAM CROOKES, F.R.S., M.R.I.

(Continued from p. 4).

Conversion of Diamond into Graphite.

I WILL now draw your attention to a strange property of the diamond, which at first sight might seem to argue against the great permanence and unalterability of this stone. It has been ascertained that the cause of phosphorescence is in some way connected with the hammering of the gaseous molecules, violently driven from the negative pole on to the surface of the body under examination, and so great is the energy of the bombardment, that impinging on a piece of platinum, or even iridium, the metal will actually melt. When the diamond is thus bombarded in a radiant matter tube the result is startling. It not only phosphoresces, but assumes a brown colour, and when the action is long continued becomes almost black.

I will project a diamond on the screen and bombard it with radiant matter before your eyes. I do not like to anticipate a failure, but here I am entirely at the mercy of my diamond. I cannot rehearse this experiment beforehand, and it may happen that the diamond I have selected will not blacken in reasonable time. Some visibly darken in a few minutes, while others, more leisurely in their ways, require an hour.

This blackening is only superficial, but no ordinary means of cleaning will remove the discolouration. Ordinary oxidising reagents have little or no effect in restoring the colour. The black stain on the diamond is due to a form of graphite which is very resistant to oxidation. It is not necessary to expose the diamond in a vacuum to electrical excitement in order to produce this change.

I have already signified that there are various degrees of refractoriness to chemical reagents among the different forms of graphite. Some dissolve in strong nitric acid; other forms of graphite require a mixture of highly concentrated nitric acid and potassium chlorate to attack them, and even with this intensely powerful agent some graphites resist longer than others. M. Moissan has shown that the power of resistance to nitric acid and potassium chlorate is in proportion to the temperature at which the graphite was formed, and with tolerable certainty we can estimate this temperature by the resistance of the specimen of graphite to this reagent.

The superficial dark coating on a diamond after exposure to molecular bombardment I have proved to be graphite,† and M. Moissan ‡ has shown that this graphite, on account of its great resistance to oxidising reagents, cannot have been formed at a lower temperature than 3600° C.

It is therefore manifest that the bombarding molecules, carrying with them an electric charge, and striking the diamond with enormous velocity, raise the superficial layer to the temperature of the electric arc, and turn it into graphite, whilst the mass of diamond and its conductivity to heat are sufficient to keep down the general temperature to such a point that the tube appears scarcely more than warm to the touch.

A similar action occurs with silver, the superficial layers of which can be raised to a red heat without the whole mass becoming more than warm.§

* A Lecture delivered at the Royal Institution, Friday, June 11th, 1897.

† *Chemical News*, vol. lxxiv., p. 39, July, 1896.

‡ *Comptes Rendus*, cxxiv., p. 653.

§ *Proc. R. S.*, vol. l., p. 99., June 1891.

This conversion of diamond into graphite is I believe a pure effect of heat. In 1880* Professor Dewar in this theatre placed a crystal of diamond in a carbon tube through which a current of hydrogen was maintained. The tube was heated from the outside by an electric arc, and in a few minutes the diamond was converted into graphite. I will now show you that a clear crystal of diamond, heated in the electric arc (temperature 3600° C.) is converted into graphite, and this graphite is most refractory.

The diamond is remarkable in another respect. It is extremely transparent to the Röntgen rays, whereas highly refracting glass, used in imitation diamonds, is almost perfectly opaque to the rays. I exposed over a photographic plate to the X rays for a few seconds the large Delhi diamond, of a fine pink colour, weighing 31½ carats, a black diamond weighing 23 carats, together with an imitation in glass of the pink diamond lent me by Mr. Streeter; also a flat triangular crystal of diamond of pure water, and a piece of glass of the same shape and size. On development, the impression where the diamond obscured the rays was found to be strong, showing that most rays passed through, while the glass was practically opaque. By this means imitation diamonds and some other false gems can readily be detected and distinguished from the true gems. It would take a good observer to distinguish my pure triangular diamond from the adjacent glass imitation.

Genesis of the Diamond.

Speculations as to the probable origin of the diamond have been greatly forwarded by patient research, and particularly by improved means of obtaining high temperatures. Thanks to the success of Professor Moissan, whose name will always be associated with the artificial production of diamonds, we are able to-day to manufacture diamonds in our laboratories—minutely microscopic, it is true—all the same veritable diamonds, with crystalline form and appearance, colour, hardness, and action on light the same as the natural gem.

Until recent years carbon was considered absolutely non-volatile and infusible; but the enormous temperatures at the disposal of experimentalists—by the introduction of electricity—show that, instead of breaking rules, carbon obeys the same laws that govern other bodies. It volatilises at the ordinary pressure at a temperature of about 3600° C., and passes from the solid to the gaseous state without liquefying. It has been found that other bodies which volatilise without liquefying at the ordinary pressure will easily liquefy if pressure is added to temperature. Thus, arsenic liquefies under the action of heat if the pressure is increased; it naturally follows that if along with the requisite temperature sufficient pressure is applied, liquefaction of carbon will be likely to take place, when on cooling it will crystallise. But carbon at high temperatures is a most energetic chemical agent, and if it can get hold of oxygen from the atmosphere or any compound containing it, it will oxidise and fly off in the form of carbonic acid. Heat and pressure therefore are of no avail unless the carbon can be kept inert.

It has long been known that iron when melted dissolves carbon, and on cooling liberates it in the form of graphite. Moissan discovered that several other metals have similar properties, especially silver; but iron is the best solvent for carbon. The quantity of carbon entering into solution increases with the temperature, and on cooling in ordinary circumstances it is largely deposited as crystalline graphite.

Professor Dewar has made a calculation as to the Critical Pressure of Carbon—that is, the lowest pressure at which carbon can be got to assume the liquid state at its critical temperature, that is the highest temperature at which liquefaction is possible. He starts from the

* *Proceedings of the Royal Institution*, Friday Evening Meeting, Jan. 16, 1880.

vaporising or boiling point of carbon, which, from the experiments of Violle and others on the electric arc, is about 3600° C., or 3874° Absolute. The critical point of a substance on the average is 1·5 times its absolute boiling point. Therefore the critical point of carbon is 5811° Ab., or, say, 5800° Ab. But the absolute critical temperature divided by the critical pressure is for elements never less than 2·5. Then—

$$\frac{5800^{\circ} \text{ A.}}{\text{PCr}} = 2\cdot5, \text{ or } \text{PCr} = \frac{5800^{\circ} \text{ A.}}{2\cdot5}, \text{ or } 2320 \text{ atmospheres.}$$

The result is that the critical pressure of carbon is about 2300 atmospheres, or say 15 tons on the square inch. The highest critical pressure recorded is that of water, amounting to 195 atmospheres, and the lowest that of hydrogen, about 20 atmospheres. In other words, the critical pressure of water is ten times that of hydrogen, and the critical pressure of carbon is ten times that of water.

Now 15 tons on the square inch is not a difficult pressure to obtain in a closed vessel. In their researches on the gases from fired gunpowder and cordite, Sir Frederick Abel and Sir Andrew Noble obtained in closed steel cylinders pressures as great as 95 tons to the square inch, and temperatures as high as 4000° C. Here, then, if the observations are correct, we have sufficient temperature and enough pressure to liquefy carbon; and if the temperature could only be allowed to act for a sufficient time on the carbon there is little doubt that the artificial formation of diamonds would soon pass from the microscopic stage to a scale more likely to satisfy the requirements of science, industry, and personal decoration.

Artificial Manufacture of the Diamond.

I now proceed to manufacture a diamond before your eyes—don't think I yet have a talisman that will make me rich beyond the dreams of avarice! Hitherto the results have been very microscopic, and are chiefly of scientific interest in showing us Nature's workshop, and how we may ultimately hope to vie with her in the manufacture of diamonds. Unfortunately the operations of separating the diamond from the iron and other bodies with which it is associated are somewhat prolonged—nearly a fortnight being required to detach it from the iron, graphite, and other matters in which it is embedded. I can, however, show the different stages of the operations, and project on the screen diamonds made in this manner.

In Paris recently I saw the operation carried out by M. Moissan, the discoverer of this method of making carbon separate out in the transparent crystalline form, and I can show you the operations straight as it were from the inventor's laboratory. I am also indebted to the Directors of the Notting Hill Electric Lighting Co. and to the General Manager, Mr. Schultz, for enabling me to perform several operations at their central station, where currents of 500 ampères and 100 volts were placed at my disposal.

The first necessity is to select pure iron—free from sulphur, silicon, phosphorus, &c.,—and to pack it in a carbon crucible with pure charcoal from sugar. Half a pound of this iron is then put into the body of the electric furnace, and a powerful arc formed close above it between carbon poles, utilising a current of 700 ampères at 40 volts pressure. The iron rapidly melts and saturates itself with carbon. After a few minutes' heating to a temperature above 4000° C.—a temperature at which the lime of the furnace melts like wax and volatilises in clouds—the current is stopped, and the dazzling fiery crucible is plunged beneath the surface of cold water, where it is held till it sinks below a red heat. As is well known, iron increases in volume at the moment of passing from the liquid to the solid state. The sudden cooling solidifies the outer layer of iron and holds the inner molten mass in a tight grip. The expan-

sion of the inner liquid on solidifying produces an enormous pressure and under the stress of this pressure the dissolved carbon separates out in a transparent, dense crystalline form—in fact, as diamond.

Now commences the tedious part of the process. The metallic ingot is attacked with hot nitro-hydrochloric acid until no more iron is dissolved. The bulky residue consists chiefly of graphite, together with translucent flakes of a chestnut-coloured carbon, black opaque carbon of a density of from 3·0 to 3·5, and hard as diamonds—black diamonds or carbonado, in fact—and a small portion of transparent colourless diamonds showing crystalline structures. Besides these, there may be carbide of silicon and corundum, arising from impurities in the materials employed.

The residue is first heated for some hours with strong sulphuric acid at the boiling point, with the cautious addition of powdered nitre. It is then well washed and allowed for two days to soak in strong hydrofluoric acid in the cold, then in boiling acid. After this treatment the soft graphite will disappear, and most if not all of the silicon compounds will be destroyed. Hot sulphuric acid is again applied to destroy the fluorides, and the residue, well washed, is repeatedly attacked with a mixture of the strongest nitric acid and powdered potassium chlorate, kept warm, but to avoid explosions not above 60° C. This ceremony must be repeated six or eight times, when all the hard graphite will gradually be dissolved, and little else left but graphitic oxide, diamond, and the harder carbonado and boart. The residue is fused for an hour in fluorhydrate of fluoride of potassium, then boiled out in water, and again heated in sulphuric acid. The well washed grains which resist this energetic treatment are dried, carefully deposited on a slide, and examined under the microscope. Along with numerous pieces of black diamond are seen transparent colourless pieces, some amorphous, others with a crystalline appearance, as I have attempted to reproduce in diagrams. Although many fragments of crystals occur, it is remarkable that I have never seen a complete crystal. All appear broken up, as if on being liberated from the intense pressure under which they were formed they burst asunder. I have direct evidence of this phenomenon. A very fine piece of artificial diamond, carefully mounted by me on a microscopic slide, exploded during the night and covered my slide with fragments. This bursting paroxysm is not unknown at the Kimberley mines.

On the screen I will project fragments of artificial diamond, some lent me by Professor Roberts-Austen, others of my own make; while on the wall you will see drawings of diamonds copied from M. Moissan's book on the Electric Furnace. Unfortunately these specimens are all microscopic. The largest artificial diamond, so far, is less than one millimetre across.

Laboratory diamonds burn in the air before the blow-pipe to carbonic acid; and in lustre, crystalline form, optical properties, density, and hardness they are identical with the natural stone.

Many circumstances point to the conclusion that the diamond of the chemist and the diamond of the mine are strangely akin as to origin. It is conclusively proved that the diamond has not been formed *in situ* in the blue ground. The diamond genesis must have taken place at great depths under enormous pressure. The explosion of large diamonds on coming to the surface shows extreme tension. More diamonds are found in fragments and splinters than in perfect crystals; and it is noteworthy that although many of these splinters and fragments are derived from the breaking up of a large crystal yet in no instance have pieces been found which could be fitted together. Does not this fact point to the conclusion that the blue ground is not their true matrix? Nature does not make fragments of crystals. As the edges of the crystals are still sharp and unabraded, the *locus* of formation cannot have been very distant from the present sites. There were probably many sites of

crystallisation differing in place and time, or we should not see such distinctive characters in the gems from different mines, nor indeed in the diamonds from different parts of the same mine.

(To be continued).

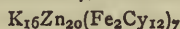
ON THE VOLUMETRIC DETERMINATION OF ZINC BY POTASSIUM FERROCYANIDE.

By L. L. DE KONINCK and EUG. PROST.

(Continued from p. 6).

IN fact, by running, to the exact precipitation point, a solution of ferrocyanide into a measured volume of zinc solution acidulated with sulphuric acid, he asserts that there is required, for 25 c.c. of zinc solution, not 12.5 c.c. of ferrocyanide, but 17.75 c.c. to 19.5 c.c., according to the acidity and concentration of the mixture.

The volume of ferrocyanide used diminishes as the acidity increases, and also with the dilution of the zinc solution. According to Zulkowski it would be quite indifferent whether the zinc solution is run into the potassium ferrocyanide or *vice versa*. By boiling the liquid, constant results can be obtained independent of the acidity, and the precipitate—which is then pulverulent instead of gelatinous—will have a constant composition.* Zulkowski recognised the end of his tests by the formation of Prussian blue, by placing side by side on a piece of filter-paper a drop of the mixture and a drop of ferric chloride. By taking note of the excess of ferrocyanide necessary to show with the ferric chloride (0.4 c.c. for each 85 c.c. of the mixture), he concludes that—



is the correct formula for the precipitate obtained by heating, and $K_{16}Zn_{12}(Fe_2Cy_{12})_5$ for that obtained in the cold, but he is not very certain of the latter.

These complicated formulæ seem at first sight improbable,—we shall show, in fact, that they ought to be rejected; the estimations or calculations on which they are based are full of errors.†

F. Regelsberger (*Zeitsch. f. Angew. Ch.*, vol. iv., p. 475, 1891), when analysing alloys of aluminium, quotes the estimation by ferrocyanide as very rapid and quite sufficiently exact.

We will here refer again to the work of Bein and Bragard. The former, who evidently had not the experience necessary to judge the value of volumetric methods to a nicety, does not speak well either of Schaffner's method or of the Galletti-Fahlberg, but prefers the gravimetric method.

Bragard gives the ferrocyanide precipitate the composition answering to the formula $K_2Zn_3Fe_2Cy_{12}$ when the reaction is at an end, in which we think he is right; but when the zinc is in excess the composition would be remedied by the formula $K_4Zn_{10}(Fe_2Cy_{12})_3$; here we think he is mistaken. According to him the precipitate would react with the nitrate of uranium used as indicator. He also interposes a sheet of filter-paper between the rod carrying the trial drop and the paper impregnated with uranium salt, and does not consider the reaction at an end while an appreciable colouration is produced on this second paper. In our opinion he thus diminishes the sensitiveness of the indicator; also, according to him, the process is not exact to less than $\frac{1}{4}$ per cent. The acidity of the liquid, he says, influences the result, but not the

dilution, of the presence of ammonia,—he evidently means salts of ammonia.

To complete our references to work which has already been published, we must again mention the Report made to the Society of Colorado. The Committee charged with examining into the methods employed in this important mining district sent to several chemists five samples of minerals, carefully selected, and analysed with every possible precaution, begging them at the same time to send, with their results, the details of the process used. All of them titrated the acid solution with ferrocyanide, using a salt of uranium as indicator. The results were as follows:—

No.	A.	B.	C.	D.	E.	F.*	G.
1.	15.31	15.39	15.66	15.08	14.62	—	14.38 14.22
2.	24.34	24.53	24.23	23.80	22.00	23.62	22.95 23.11
3.	10.76	10.83	11.88	10.69	10.50	11.07	8.58 9.20
4.	6.42	6.58	8.73	6.85	6.30	6.89	5.24 5.64
5.	16.14	16.46	15.86	15.90	15.37	16.08	13.40 12.84

* Average of four concordant assays. No. 1 was not analysed.

The actual results obtained gravimetrically were:—

14.64 24.11 10.71 6.31 16.09.

The samples 1 and 2, but especially No. 1, contained cadmium in appreciable quantity, which will account for the generally high results, this metal not having been eliminated. It is worthy of remark that they do not as a rule use hydrosulphuric acid; when the mineral contains copper they precipitate it by means of granulated lead, which is well shaken up in the acid solution.

We may conclude from these results that the process will furnish exact results, so long as it is carried out under specified conditions and with the necessary care.

III. Reaction of Ferrocyanide of Potassium with Zinc Salts in Acid Solution.

As we have seen, there is very little accord as to the composition of the precipitate, which is formed in an acid zinc solution, on the addition of ferrocyanide.

All admit that if ferrocyanide be added in sufficient quantity for it to be in slight excess, and to be detected in the mixture by a uranium salt, or by other means, the quantity employed is notably more than that which is necessary theoretically to produce a simple zinc ferrocyanide, $Zn_4Fe_2Cy_{12}$. Nevertheless some chemists give this formula to the precipitate and explain away the difference, although it is considerable, by impurities in the substance used.

The authors of this paper, who have studied ferrocyanides, have long been aware that the zinc precipitate obtained by an excess of ferrocyanide is a double salt, $K_2Zn_3Fe_2Cy_{12}$ (Mosander, *Handw. d. Reinen u. Angew. Chem.*, p. 867, 1848; F. Reindel, *Polyt. Journ de Dingler*, vol. cxc., p. 395, 1869; G. Wyruboff, *Ann. de Chim. et de Phys.*, Series 5, vol. viii., p. 444, 1876); that has not prevented many other analysts attributing to it much more complicated formulæ. This question of the composition of the precipitate,—that is to say, the relation between the zinc and the ferrocyanide,—being of the first importance to us, it was imperative to decide the point definitely, once and for all; in this we have been successful. Our work has settled the point in dispute.

In fact, from the first titrations done by us, to get some idea of the true value of the ferrocyanide process, as described by most writers (direct titration in acid solution), we found, by running a decinormal solution* of ferrocyanide into 25 c.c. of a similarly decinormal solution of zinc chloride,† made slightly acid and diluted with water

* We shall see later that this transformation is of great importance.

† The author must have made a mistake in his calculations; the reaction, in presence of ferrocyanide of potassium in excess, is very sharp, and the relation between the potassium and the zinc is 2 to 3, or 8 to 12, and not 16 to 12.

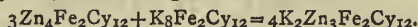
* The molecular weight of $K_4Fe_2Cy_{12} \cdot 6H_2O$, being 843.52, the normal weight is represented by $\frac{1}{10}$ of this figure, viz., 84.352. Our decinormal solution was prepared by dissolving 52.72 grms. of pure ferrocyanide in 1 litre of water.

† Obtained by dissolving 16.2775 grms. of pure zinc in the smallest possible quantity of hydrochloric acid, and then diluting to 1 litre.

to 150 c.c., that, working with from 26 to 28 c.c. of ferrocyanide, we obtained, by testing immediately with uranium salts,* an appreciable brown colouration, which does not get stronger by the addition of ferrocyanide, which is further less sensitive as we increase the time from the addition of the titrate and the touch test, and eventually disappears if this time be sufficiently prolonged. But with from 33·3 to 33·5 c.c. the test becomes more and more distinct, and that no matter what time elapses before the addition of the ferrocyanide. These facts can be explained in several different manners.

The most plausible which first occurred to us is the following:—When the ferrocyanide is added to the zinc solution, a ferrocyanide of zinc, $Zn_4Fe_2Cy_{12}$, is first formed, so long as the zinc salt is in excess. The formula, $4ZnCl_2 + K_3Fe_2Cy_{12} = 8KCl + Zn_4Fe_2Cy_{12}$, would therefore show what takes place during the first part of the reaction,—that is to say, in our experiment, up to the addition of 25 c.c. of ferrocyanide.

By continuing to add this reagent, the alkaline ferrocyanide would then reach more or less slowly with the zinc precipitate, forming the double ferrocyanide, $K_2Zn_3Fe_2Cy_{12}$, according to the formula—



This reaction is not immediate; the mixture will react with the nitrate of uranium, while neither the zincic ferrocyanide nor the double ferrocyanide do.

Certain tests, to which we shall refer later on, incline us to the belief that the double cyanide is really formed in the first instance; that it, being gelatinous at first, can in this condition react with the uranium salt so long as it is not in presence of a large excess of zinc; but that it undergoes a molecular transformation which shows itself by passing from the translucent gelatinous state to a more coherent granular state, in which it will not act with the indicator. Perhaps both theories are correct,—that is to say, the phenomena may be produced simultaneously. It is in any case proved that the indicator does not show, in a definite manner, that when the volume of ferrocyanide used, corresponds sensibly to the formation of the double salt, and that this latter, the same as with the simple zincic ferrocyanide, if there be one formed, will not react with the nitrate of uranium after a sufficient digestion. It is equally shown that an excess of ferrocyanide of potassium does not combine with the double salt, $K_2Zn_3Fe_2Cy_{12}$, at least not under the conditions of our experiments, which were carried out with a deminormal solution of zincic chloride and a solution, $\frac{1}{2}$ normal for zinc,† of ferrocyanide.

We here give the result. In each case we used 25 c.c. of $ZnCl_2$, diluted with 100 c.c. of water and acidulated with hydrochloric.

A. Add 29 c.c. of ferrocyanide. The zinc is now in excess, even allowing the formation of $Zn_4Fe_2Cy_{12}$, which would require 37·5 c.c. for complete precipitation. The precipitate, gelatinous at first, becomes gradually white and flocculent; it collects and deposits in less than fifteen minutes.

B. Add 38 c.c. of ferrocyanide, that is, 0·5 c.c. more than the quantity necessary to precipitate the zinc entirely, as $Zn_4Fe_2Cy_{12}$. The precipitate behaves in exactly the same way as in the preceding experiment. In the solution, cleared by sedimentation, a fresh addition of ferrocyanide produces an abundant precipitate: this proves the presence of zinc in solution, and it therefore follows that the first precipitate is not entirely composed of $Zn_4Fe_2Cy_{12}$,—if, indeed, it contains this compound. As the first precipitate, A, behaves exactly like the second one, we can only conclude that it also, in spite of the notable excess of chloride of zinc, is not $Zn_4Fe_2Cy_{12}$.

C. Add quickly 50 c.c. of ferrocyanide, that is, the

theoretical amount necessary to precipitate all the zinc as $K_2Zn_3Fe_2Cy_{12}$.

If tested immediately, the mixture gives a strong brown colouration with nitrate of uranium. If filtered at once, the mixture yields at first an absolutely limpid filtrate, which gives no colouration with uranium; it is therefore free from potassic ferrocyanide. After a few moments the filtrate becomes very cloudy, and finally becomes clear again. Neither the cloudy filtrate nor the final clear filtrate gives any colour with the indicator. A small quantity of the unfiltered mixture, put on one side, remains milky, and reacts very feebly with nitrate of uranium.

D. Add quickly, while agitating, 50 c.c. of ferrocyanide. An immediate test of the mixture with nitrate of uranium gives an intense brownish red colouration; after half a minute, a colour of "café-au-lait;" after a minute, a clear coffee-colour; after another half minute, a very feeble reaction; after two minutes, nothing at all. Then add 5 c.c. of ferrocyanide; the mixture will again show an intense brown colour when tested with uranium; the same result will be obtained after an hour, or even two hours.

(To be continued).

BASIC SALTS OF CADMIUM.

By M. TASSILLY.

THE action of metallic oxides, on the corresponding haloid salts, has enabled me to prepare two new compounds of cadmium, an oxybromide and an oxyiodide.

These bodies were obtained by heating a concentrated solution of bromide or iodide in the presence of oxide of cadmium, up to 200°, in a sealed tube. The quantities obtained were very small. These new bodies are distinctly crystalline and act on polarised light.

Analysis has given, for the oxyiodide, the formula CdI_2, CdO_3, H_2O .

	Found.	Calculated.
Iodine ..	46·45	46·35
	46·62	"
Cadmium ..	41·07	40·87

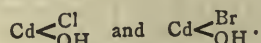
This compound is only slightly attacked by water. At 120° it does not vary in weight, either in nitrogen or in dry air freed from carbonic acid.

With parallel rays of light the crystals show extinction parallel to the longitudinal axis. In converging light we observe a lemniscate. The crystal is doubly refracting at very wide axes.

The oxybromide is in very small crystals, answering to the formula $CdBr_2, CdO_3, H_2O$; it also acts on polarised light.

	Found.	Calculated.
Bromine ..	36·44	35·24
Cadmium ..	48·90	49·33

I would recall the fact that M. Schulten (*Comptes Rendus*, vol. cvi., p. 1674) obtained both an oxychloride and an oxybromide, by the action of marble on chloride, or bromide of cadmium, at about 200°, in a sealed tube, both bodies being crystalline and answering respectively to the formulæ $CdCl_2, CdO, H_2O$ and $CdBr_2, CdO, H_2O$. Their stability when heated induced him to attribute to these bodies the constitutions—



M. Habermann (*Monatshette*, vol. v., p. 432, and *Bull. Soc. Chim.*, Ser. 2, vol. xlv., p. 122) obtained an amorphous oxychloride of the same formula by adding ammonia to a cold solution of chloride of cadmium as long as a precipitate was formed. It was then boiled, let stand, filtered, and the precipitate dried over quicklime under a bell-glass.

* One per cent solution of nitrate of uranium.

† We mean to infer, by the expression normal for zinc, a solution which will precipitate, volume for volume, a normal solution of zinc in the form $K_2Zn_3Fe_2Cy_{12}$.

By acting in the same manner on the bromide and the iodide, I obtained two compounds corresponding to definite formulæ; but the preparation of these bodies necessitates certain precautions. In fact, if we pour strong ammonia into solutions of chloride and bromide at 1/5th and of iodide at 1/7th strength, we obtain at the same time a basic salt and an ammoniacal salt which are very difficult to separate.

Working with more dilute solutions (1/10th) and adding ammonia equally diluted (ammonia at 22°, to which is added its own volume of water), we obtain first a precipitate of the basic salt, and some hours after decantation there forms in the mother-liquor crystals of the ammoniacal salt. One can even prevent altogether the formation of ammoniacal salts, by carefully limiting the proportion of ammonia.

The ammoniacal salts obtained under these conditions agree with the formulæ $\text{CdCl}_2 \cdot 2\text{NH}_3$; $\text{CdBr}_2 \cdot 2\text{NH}_3$; $\text{CdI}_2 \cdot 2\text{NH}_3$.

These bodies are identical with those obtained by dissolving the corresponding salts of cadmium in ammonia.

The oxybromide, $\text{CdBr}_2 \cdot \text{CdO} \cdot \text{H}_2\text{O}$, and the oxyiodide, $\text{CdI}_2 \cdot \text{CdO} \cdot \text{H}_2\text{O}$, gave on analysis the following results:—

Oxybromide.

	Found.	Calculated.
Bromine.. ..	37'17	38'29
Cadmium . . .	54'23	53'58

Oxyiodide.

Iodine	49'77	49'63
Cadmium . . .	43'54	43'75

The basic salts of cadmium obtained by precipitation are decomposed by water.

I would here further remark that, in the action of ammonia on the salts of cadmium in solution, the proportions of basic salts obtained decrease from the oxychloride to the oxyiodide, while, on the contrary, the proportions of ammoniacal salts increase from the chloride to the iodide.

The chloride would thus appear to give a basic salt more easily than an ammoniacal one; the inverse should be the case for the iodide.

The thermo-chemical study of these various bodies, which I am now occupied with, will without doubt give the reason of these facts.

In finishing, it may be remarked that the basic salts of cadmium are always formed in equal molecules, contrary to what happens in the case of the salts of zinc, which are liable to fix a variable number of molecules of oxygen, giving rise to a number of salts not corresponding to a general type, as occurs with certain other metals.—*Bull. Soc. Chim. de Paris*, Series 3, vol. xvii.—xviii., No. 12.

HYPOIODOUS ACID AND HYPOIODITES.*

By R. L. TAYLOR, F.C.S.

Hypoiodites.

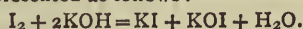
It appears to have been always considered very doubtful whether hypoiodous acid has ever been prepared at all, and many chemists are hardly willing to recognise hypoiodites as very definite compounds. The information one can obtain about these bodies is very vague and indefinite, and in some respects contradictory.

My investigation was originally undertaken with the object of isolating hypoiodous acid, but the following experiment led me to include hypoiodites as well. I had found that a solution of iodine in water acted in many respects very much better than any other solution, or

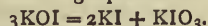
than the solid substance, and trying the effect of adding a little alkali to some of this aqueous solution, I was astonished at the particularly definite character of the solution obtained, and especially at its bleaching action, and felt sure that this remarkable solution could not be generally known, or else hypoiodites would certainly have met with better recognition than they have hitherto received.

So far as I am aware, the most important papers on hypoiodites have been those by Schönbein (*Journal für Praktische Chemie*, 1861, p. 387), and by G. Lunge and R. Schoch (*Berichte*, xv., p. 1883) on Calcium Hypoiodite. The more important of these is that by Schönbein, and in the first part of this paper I shall describe some of Schönbein's experiments, with others which I have performed and which confirm and extend his results. I shall refer to the work of Lunge and Schoch afterwards. I find that Schönbein, in his experiments, used the very solution which I have already mentioned as giving such remarkable results, that is, iodine dissolved in water. Unfortunately, however, Schönbein's paper has been badly summarised in all the standard dictionaries and works on chemistry, and this important point is not usually mentioned. Schönbein's paper is one of a series. He had been trying experiments on the action of chlorine water and bromine water upon dilute ammonia, and then naturally passed on to iodine, using that substance also in solution in water. Such a solution is very dilute, being at the most only about one part in 5000; but this solution, in many respects, gives more definite results than any other.

Schönbein first described the action of ammonia upon iodine water, whereby the liquid was decolourised, and a solution obtained which bleached indigo, just as the liquids produced by the action of ammonia upon chlorine water and bromine water did. He found, further, that the solution gave a deep blue colouration with a mixture of starch-paste and potassium iodide, and even with starch-paste alone. Left to itself the liquid lost these peculiarities, more quickly at high than at low temperatures, and almost instantaneously when boiled. He then found that similar results were obtained with potash solution, and that both solutions were decomposed by hydrogen peroxide, with manifest liberation of oxygen. He also pointed out that the solutions smelt of saffron. He not unnaturally concluded from these results that the liquids contained hypoiodites, and that the action of iodine upon the alkalis was similar to the action of chlorine and bromine, and might be represented as follows:—



He also concluded that, as the liquids lost their bleaching power, they gradually changed into iodide and iodate, according to the following equation:—



One thing which seemed to puzzle him very much was that the liquids gave a blue colour with starch alone, even when he added potash in the proportion of two equivalents to one of iodine. He thus made the liquid strongly alkaline, and capable, as he said, of taking up more iodine; and he argued, therefore, that there could not be any free iodine present in the excess of potash, and that hence the blue colour could not be due to iodine. In addition he pointed out that the liquid was almost, if not quite, colourless. He found, however, that the addition of potassium iodide turned the liquid brown again, manifestly owing to the liberation of iodine. I shall show further on that these results are easily explained.

I may mention that in all my experiments the iodine used was carefully purified by Stas's method, and that the indigo was a solution of indigo carmine in water.

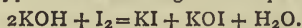
As has been already mentioned, the liquids produced by the action of alkalis upon aqueous iodine have a most energetic bleaching action upon indigo; they also bleach cochineal and logwood, but not litmus. In bleaching

* From *Memoirs and Proceedings of the Manchester Literary and Philosophical Society*, vol. xli., Part III.

indigo they are much more active than either a solution of chlorine or of bleaching powder of anything like the same strength; in fact, compared with Schönbein's solutions, chlorine and hypochlorites may be described as very sluggish.

Borrowing, with some modifications, a method described by Lunge and Schoch in their paper, I attempted, by means of a standard solution of indigo carmine, to ascertain the strength of the bleaching liquids, in order to find, if possible,—assuming that the reaction goes as Schönbein suggested, and as the corresponding reaction with chlorine and bromine are well known to go,—the amount of iodine converted into what one may call "bleaching iodine." After many attempts I found that the best results were obtained by standardising the solution of indigo carmine against a dilute solution of chlorine, which had been titrated against a standard iodine solution by means of potassium iodide and sodium thiosulphate in the usual way. The aqueous iodine solution was also standardised against the same standard solution of iodine. The amount of iodine present in the aqueous solution was usually from 0.17 to 0.22 grm. per litre. One of the difficulties experienced in standardising the solutions was due to the end-reaction with chlorine water and the indigo solution being exceedingly slow. No such difficulty, however, was anticipated with the iodine bleaching solutions, the end-reaction with these being apparently sharp and distinct.

The method employed was to take a measured volume (usually 20 c.c.) of the aqueous iodine solution, to add one or two drops of potash or soda, and then immediately run in the standard indigo carmine until there was a distinct green colour. (The indigo solution is bleached to a slightly yellow liquid, and this of course becomes green as soon as an excess of indigo is added). For a long time the results were unsatisfactory. The bleaching power of the solutions seemed to vary in an extraordinary manner. Frequently the results obtained gave 90 and 95 per cent of the iodine converted into "bleaching iodine," and then, in another experiment, with the same solution of iodine, the bleaching action, without any apparent reason, ran up to 30 or even 40 per cent above the theoretical amount,—that is, above the amount which it ought to be if the whole of the iodine used had been converted into iodide and hypoiodite according to the equation—



Similar anomalous results were obtained when solutions of bleaching powder or of sodium hypochlorite were used instead of chlorine water. These extraordinary results were ultimately found to be due to a very strange action on the part of the indigo, an action of which I can at present offer no explanation. The excess of bleaching action upon the indigo is not permanent; on standing for a minute or two the blue colour returns. This of course is not the case with what I may call the genuine bleaching action. If 1 c.c. of indigo solution in excess of what is permanently bleached be added, although there appears to be no indication of when the end-point is being passed, on standing for a minute or two a blue colour appears. I further found that this curious temporary bleaching action only occurs when a large excess of alkali (in comparison with the amount of iodine present) has been used.

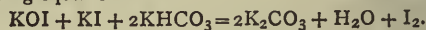
Now, of course, it was possible to determine the amount of permanent bleaching action. The following example is one out of a great many experiments which I made:—

Twenty c.c. of the aqueous iodine solution (=0.0035 iodine), after the addition of alkali, bleached 15 c.c. of standard indigo solution, 1 c.c. of which (titrated with standard solution of chlorine) corresponded to 0.000228 of iodine, so that the amount of iodine indicated by the bleaching action was $0.000228 \times 15 = 0.00342$, which was practically the whole of the iodine. The solutions used are extremely dilute, but there is really no difficulty in

making estimations which will be accurate to within 2 or 3 per cent. The general result of these experiments is that 95 per cent of the iodine in Schönbein's solutions undergoes the reaction represented by the equation—

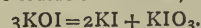


These results are amply confirmed by an altogether different method,—one which was used by A. Schwicker (*Zeit. Physikal. Chem.*, xvi., 303-314) in an investigation which he has recently made on the reaction velocity of potassium hypoiodite. He takes advantage of the fact that potassium bicarbonate will decompose a mixture of hypoiodite and iodide, with liberation of iodine. He also uses a little soda-water, the carbonic acid in which is intended to convert any liberated potash into the bicarbonate. The bicarbonate apparently decomposes the mixture of hypoiodite and iodide, with formation of normal carbonate and liberation of iodine, according to the following equation:—



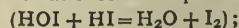
With my dilute solutions, I find that it answers just as well to run into the liquid, which is always sufficiently alkaline, a small quantity of soda-water. This immediately liberates the iodine, which can now be estimated by means of a centi-normal solution of sodium arsenite. Carbonic acid does not decompose potassium iodate, so that this method may be employed in all mixtures of hypoiodites, iodates, and iodides. In one determination by this method 97 per cent of the iodine originally used was liberated on the addition of the soda-water. We may therefore conclude that when potash acts upon iodine-water there is practically no iodate formed.

As Schönbein pointed out, the solutions are very unstable. I have made a number of experiments upon the rate at which the change occurs, estimating this by the diminution in bleaching power. I find that the presence of excess of alkali makes the solution more stable; but even then a solution loses half its bleaching power on standing, in the dark, for four hours. In twenty-four hours 75 per cent of the bleaching power goes. If a much smaller amount of alkali is used half the bleaching power goes in an hour. On heating the solutions they alter very rapidly, and every bleaching liquid of this kind which I have prepared loses its bleaching power entirely if boiled for three or four minutes. As Schönbein assumed, this loss of bleaching power is doubtless due to a change of the hypoiodite into iodide and iodate,—

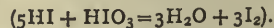


As mentioned above, Schwicker has recently investigated the rate at which the above change occurs at the ordinary temperature with different proportions of iodine and potash present. The results do not appear to have been altogether satisfactory. But he used iodine dissolved in potassium iodide, and there is no doubt that the latter would affect the results materially. Probably better results would be obtained by the use of a solution of hypoiodite made from hypoiodous acid, which would not contain any iodide at all.

I may refer here to the fact that whether the liquid contains iodide and hypoiodite, or iodide and iodate, the addition of an acid at once liberates the whole of the iodine; in the one case hypoiodous and hydriodic acids are liberated, which at once decompose each other—



in the other, hydriodic and iodic acids are similarly liberated, and in the exact amounts needed to decompose each other—



I have made similar bleaching solutions by using lime-water and baryta-water with aqueous iodine, and in nearly all respects these resemble Schönbein's solutions, there being perhaps a little difference in their stability in favour of the sodium and potassium compounds. They are all decomposed on boiling.

I have further found that, by using a little very finely-divided (preferably precipitated) iodine with the aqueous iodine, and then adding the alkali, very much stronger solutions may be prepared. A solution made in this way bleaches large quantities of indigo, and gives further reactions which add very strongly to the evidence that these solutions contain hypoiodites. Thus they give a black precipitate (on standing) with a cobalt solution; an immediate dark brown precipitate with a solution of a manganous salt; and with lead salts a precipitate which manifestly contains a considerable amount of the brown peroxide of lead. Also these strong solutions give an immediate and copious evolution of oxygen with hydrogen dioxide. In these reactions the solution acts exactly as the corresponding hypochlorites and hypobromites do. The dilute solutions made with aqueous iodine naturally do not give these reactions so satisfactorily unless large quantities are used. On the other hand, the stronger solutions would not be so suitable for the quantitative experiments as the more dilute ones.

The solution made with iodine-water and not too much alkali gives with nitrate of silver a precipitate which is quite distinct from the ordinary precipitated hydrate of silver, having a sort of dark buff colour. Of course the precipitate must contain silver iodide and probably also some hydrate, as the original liquid must of necessity be somewhat alkaline; but it probably also contains some silver hypoiodite. If the liquid is poured off or filtered off from the precipitate, it is found to have completely lost its bleaching power. On the other hand, if the precipitate is treated with a dilute acid, part of it dissolves up, leaving the yellow iodide of silver, and at the same time the solution acquires bleaching properties, though not to anything like the extent that would correspond to a complete transformation of the hypoiodite into a silver salt, and then to hypoiodous acid. In the two transformations a large amount of the hypoiodite is evidently decomposed.

I have already mentioned that Schönbein was greatly puzzled to account for his bleaching solutions giving a deep blue colour with starch alone. Lunge and Schoch in their paper suggested that some iodine probably existed in the liquid in combination with potassium iodide. But a much more reasonable explanation had already been supplied by the experiments of E. Lenssen and J. Löwenthal (*Journ. für Praktische Chemie*, 1862, p. 245), who found that sodium iodide and hypoiodite decompose each other, liberating iodine, and that the amount of alkali required to react with free iodine was greater when potassium iodide was present than when there was no iodide. They practically stated that the reaction—



is a balanced one, and that the addition of potassium iodide reverses the action, which now produces potash and free iodine.

It follows that the amount of alkali required to complete the above reaction must be greater than that represented by the equation. I have added varying amounts of a standard solution of soda to the same amount of aqueous iodine. With one equivalent of alkali to one of iodine the solution is distinctly yellow, and gives a deep blue colour with starch; with two equivalents of alkali the liquid is a very pale yellow, and the colour with starch is much less intense; with three equivalents the liquid appears colourless, and gives only a slight colour with starch, so that apparently the reaction is all but complete, and with four equivalents it is quite complete.

It is probable that the character of this reaction has something to do with the comparative failure to obtain bleaching solutions when using iodine dissolved in potassium iodide. It may also help to explain the fact that in the action of ozone upon potassium iodide the development of free iodine may proceed to quite a remarkable extent, considering that its liberation must be accompanied by the formation of an equivalent amount

of potash. It is clear, however, that, as there is always a very large excess of potassium iodide present, this must tend to prevent the formation of any but the smallest amount of hypoiodite.

In 1882 the paper by Lunge and Schoch on Calcium Hypoiodite appeared. The authors criticised Schönbein's work at some length. They objected to the importance which Schönbein appeared to attach to the fact that his solutions gave an evolution of oxygen with hydrogen peroxide, pointing out that a mixture of potassium iodide and iodate does the same thing. There is a certain amount of weight in this objection, but not much. It is quite true that a mixture of iodide and iodate does evolve oxygen with hydrogen dioxide, but only either on standing or when gently warmed; whereas, as I have already pointed out, the stronger hypoiodite solutions which I have prepared give an immediate violent effervescence on the addition of the peroxide. Schönbein's dilute solutions certainly do not give oxygen anything like so rapidly as these stronger ones, but still much more rapidly than a mixture of potassium iodide and iodate. I still consider, with Schönbein, that the immediate evolution of oxygen with hydrogen peroxide is a valuable indication that these solutions contain hypoiodites.

Lunge and Schoch prepared their "hypoiodite of calcium" by rubbing together for some time iodine with a large excess of lime and a comparatively small amount of water, allowing to stand for some hours, and then diluting with water. They thus obtained a solution which apparently resembled Schönbein's solutions in many respects, but gave "with cobaltous nitrate a green precipitate—no black peroxide." It bleached cochineal, logwood, and indigo carmine, just as Schönbein's solutions do.

The authors attempted to estimate the bleaching strength of the solution by means of a standard solution of indigo carmine, standardised against a dilute solution of bleaching powder, the strength of which was estimated by means of a standard solution of sodium arsenite. But they could not succeed in measuring the bleaching power of their iodine-lime solution directly, because towards the end the decolourisation was so extraordinarily slow. They therefore added an excess of indigo solution, and allowed to stand for fifteen minutes; then excess of bleaching powder solution was added, and this excess finally brought back by a drop or two of sodium arsenite solution. In this way they estimated that in their solution 14.6 per cent of the total iodine present existed as "bleaching iodine." They further stated that the solution, kept in the dark, gradually lost its bleaching power, but that only 76 per cent of the bleaching action had disappeared at the end of twenty-three days. They also tried the effect of heating the solution, and found, on one occasion, that when boiled for one hour 52 per cent of the bleaching power had disappeared. In another experiment a sample was boiled for seven hours, and then only lost 53 per cent of its bleaching power!

It is evident that there are some irreconcilable discrepancies between these results and mine. In the first place, I never found any difficulty in estimating the bleaching power of a solution directly, except in the case where the bleaching is not permanent. Secondly, my solutions gave black precipitates with cobalt; and, in the next place, every bleaching solution that I have made is decomposed completely by boiling for, at most, four minutes. Judging from the analogous bodies, hypochlorites and hypobromites, and from the instability of hypoiodites on merely keeping them in the dark, it is inherently highly improbable that any hypoiodite could stand being boiled for seven hours! I have prepared what I should call calcium hypoiodite by adding lime-water to aqueous iodine, and it decomposes completely when boiled for three minutes. Whether the complicated method adopted by Lunge and Schoch for estimating the bleaching action has anything to do with these discrepancies I am not prepared to say, but it seems quite

plain that if Schönbein's solutions consist of hypiodites, then Lunge and Schoch's solution does not. I have tried to repeat Lunge and Schoch's experiments, following their directions, and have obtained a bleaching liquid which acts practically like Schönbein's solutions; that is, there is little or no difficulty in estimating the bleaching power directly, and it loses its bleaching power completely when boiled for a few minutes. If, also, as I should recommend, the iodine and lime are rubbed together with water, and then diluted *immediately*, instead of, as they recommend, allowing the mixture to stand for several hours, a solution is obtained three or four times as strong, which gives a dark brown precipitate with cobalt; but this also is decomposed completely when boiled for a few minutes. It also gradually decomposes when kept in the dark, and a sample tested on one occasion, after being left for three days, had lost entirely its bleaching power.

Since the appearance of Lunge and Schoch's paper there have been occasional references to hypiodites in other papers. Thus C. Lonnes (*Zeit. Anal. Chem.*, xxxv., 409-436) has pointed out that the conversion of iodine into an iodide and an iodate by an alkali is not immediately complete, part remaining uncombined, and part being converted into hypiodite, and that the hypiodite has greater stability in presence of excess of alkali.

Chattaway (*Chem. Soc. Journ.*, lxi., p. 1572) has stated that in several of the decompositions which the so-called "nitrogen iodide" undergoes, hypiodites are produced.

Quite recently (*Proc. Roy. Soc. Edin.*, xxi., 235) Dr. J. Walker and S. A. Kay, B.Sc., have published a paper on the so-called "Magnesium Hypiodite," a brown substance formed by the union of magnesia, either wet or dry, with free iodine, and which has sometimes been supposed to be magnesium hypiodite. They conclude, however, that it is simply a case of absorption of iodine, without any chemical combination. They find that this brown precipitate is produced when potash is added to a solution of iodine in potassium iodide until the iodine just disappears, and then a solution of magnesium sulphate is added, magnesium hydrate being precipitated, and iodine manifestly liberated. They have concluded from this that, as pointed out above, the reaction between iodine, potash, potassium iodide, and water, is a balanced one.

(To be continued).

THE PREPARATION OF ZINC ETHYL.

By ARTHUR LACHMAN.

Of all the methods heretofore proposed for the preparation of zinc alkyls, the Gladstone-Tribe copper-zinc couple (*J. Chem. Soc.*, 1879, 570), gives by far the best results. As originally given, however, the directions are open to two practical objections. First of all, it is not always an easy matter to secure the zinc filings of necessary fineness. And secondly, while the careful fusion of the filings with the reduced copper offers no difficulties when carried out with 10-grm. lots, as usually taken by the authors, it is practically impossible to prevent larger quantities from melting completely and forming a worthless solid ingot on cooling.

A form of zinc available in every laboratory is zinc dust. On removing the coating of oxide by heating in hydrogen for a short while, it was found that 50 grms. each of the metal and ethyl iodide reacted within ninety minutes, and gave 14 grms. of zinc ethyl (72 per cent of the theoretical yield). When this metal is coupled with copper, by reducing an intimate mixture of zinc dust and finely powdered copper oxide, the time of reaction is shortened to thirty minutes, and the yield increased to nearly 90 per cent. Different varieties of zinc dust give

different yields (ranging from 70 per cent upward); this probably finds its explanation in the phenomena discussed by H. Wislicenus (*J. prakt. Chem.* (2), liv., 18 ff). The actual quantities of materials taken also exercise an influence, the yield being somewhat smaller with larger quantities (100 grms. or more). I have not attempted to ascertain the cause of this.

The details of the process are extremely simple. Zinc dust and copper oxide are mixed in the ratio of 100 to 12, loosely filled into a combustion tube, and a current of hydrogen passed over the heated mixture for about twenty minutes. In order to prevent the condensation of moisture in the tube, with the consequent caking of the mass, it is best to heat the tube along its whole length at once. A dull red heat seems to give the best results; the nearer the melting-point of zinc the better, I presume. If the temperature be kept too low, the metals do not alloy well, and the results approach those given by zinc dust alone. I have usually employed equal quantities of ethyl iodide and metal couple, sometimes less metal; perhaps the yield might be increased by excess thereof. For further directions the article of Gladstone and Tribe had best be consulted. In place of the oil-bath for heating the zinc ethiodide, the Babo air-baths may be substituted, as being cleaner and more expeditious; if a small flame be placed under them at first, there is no danger of breaking the flask. By employing two sets of apparatus, and working up about 100 grms. at a time, the process may easily be made continuous, and several hundred grms. of zinc ethyl prepared in a single day. Mr. F. M. Tschirner, who has manufactured nearly 300 grms. for me, found no difficulty in obtaining 150 grms. in an afternoon.

It is customary to preserve zinc ethyl in sealed tubes. This is awkward where small and varying quantities are frequently needed. Small Erlenmeyer flasks, with perfectly straight sides beginning at the very mouth, greatly facilitate the operation of pouring out the liquid, and when well corked form perfectly safe receptacles. Instead of requiring an assistant to keep a stream of carbon dioxide upon the mouth of the flask when transferring zinc ethyl, it is much more convenient to conduct all such manipulations under a small, inverted funnel, through which the gas is rapidly passing.

It had been my intention to compare the above described modification of the copper-zinc couple with the original, chiefly with respect to its reducing powers, but the recent article of H. Wislicenus (*loc. cit.*) has made this unnecessary. Qualitative tests show that the new form reads in much the same manner with alkyl and alkylene haloids as the older. I have attempted to use ethyl bromide in place of the expensive iodide, but without encouraging results. Methyl iodide reacts as readily as the ethyl compound, though the yield of zinc methyl was not determined.—*American Chemical Journal*, xix., No. 5.

A SIMPLE TEST FOR THE HALOGENS IN ORGANIC HALIDES.

By J. H. KASTLE and W. A. BEATTY.

In connection with some work on the displacement of bromine and iodine from their organic compounds, we had frequent occasion to test for the halogens in their organic derivatives, and, while a great many methods for conducting such test were to be found in the literature, few, if any of these, could be conducted rapidly; and many involved the use of rather large amounts of substances, such as lime, soda-lime, &c., which are not readily obtained free from chlorine. It is unnecessary in this connection to enumerate the many tests which have been proposed for these elements in their organic compounds; most of them are already familiar to chemists,

and nearly all of them are described in Beilstein's "Handbuch" and in Allen's "Commercial Organic Analysis."

Appreciating the need therefore of a simpler and rapid test whereby chlorine, bromine, and iodine could be recognised with certainty in their most stable and volatile organic combinations, it occurred to one of us (Kastle) to heat such compounds with a mixture of silver and copper nitrates, thereby oxidising the organic matter and holding back any halogen present by means of silver. On experiment it was found that excellent results could be obtained with this mixture, which can always be obtained free from any traces of chlorine, bromine, or iodine, by dissolving the metals in pure nitric acid and evaporating to crystallisation. In the case of non-volatile substances the test is most easily conducted as follows: A small quantity of a substance to be tested, usually about 0.1 gm., is placed in the test-tube, along with about 0.5 gm. of the mixed nitrates and a few drops of water. The tube is then gradually heated in the flame of the Bunsen burner until the nitrates are completely decomposed; the temperature never being allowed to rise higher than low red heat. The tube, in which some reduced copper is often seen, is allowed to cool, a little water and dilute sulphuric acid poured on the contents of the tube and a few pieces of metallic zinc added. After five or ten minutes, during which time any halogen compound of silver is reduced, the contents of the tube are filtered and a solution of silver nitrate added to the filtrate, together with a little dilute nitrate acid, when the chloride, bromide, or iodide of silver will be precipitated, if either of these halogens was present in the substance tested.

Excellent tests for chlorine, bromine, and iodine were obtained in this way in the following substances: Dibromonaphthalene, eosine, benzene dichlorosulphonamide, *p*-bromobenzene-sulphonamide, tribromophenol, potassium *p*-bromobenzenesulphonate, monobromacetanilide, sozoiodol (Merck), barium *p*-iodobenzenesulphonate. With very volatile substances such as chloroform, it was found necessary to conduct the test in a tube about six inches long by one-quarter inch internal diameter, closed at one end, and bent twice at right angles at equal distances along the tube, so as to form a tube having somewhat the shape of the letter S. In making the test about $\frac{1}{4}$ c.c. of the volatile compound to be tested was introduced into the closed end of the tube, and the tube so clamped as to give its open end an upward slant. About 0.5 gm. of the dry mixed nitrates was then placed in the bend of the tube farthest removed from the closed end; after which the parts of the tube containing the substance to be tested and the nitrates were heated alternately, the former portion very gently, so as not to drive out the volatile substance before it could be oxidised by the decomposing nitrates. The heating is continued until all the nitrates are decomposed, and all of the substance to be treated volatilised, at the end of which time the tube is broken and the fragments, containing the oxides of copper and silver, placed in a test-tube with a small quantity of water and sulphuric acid and a few small pieces of zinc. The test is then completed in the manner described in the above. The following substances were tested in the closed S-shaped tubes with excellent results in every case:—

Chloroform, isopropyl bromide, butyl bromide, ethyl chlorocarbonate, propyl bromide, propyl iodide, bromoform, ethylidene chloride, propyl chloride, monobromobenzene, dibromobenzene, ethylene bromide, monochloroacetic acid, benzoyl chloride, dibromosuccinic acid.

It is believed that this test as applied both to volatile and to non-volatile organic halides has in it many points of advantage over those which have been proposed for this purpose; in the first place the test is rapid and certain in its result, and by means of it we can judge not only of the presence or absence of the halogens, but it can also be determined at a glance whether it is chlorine, bromine, or iodine that is present in the organic compound. Secondly, only very small quantities of sub-

stances are required to make the tests, and the few simple reagents needed may be obtained free from any traces of halogen without the least difficulty.—*American Chemical Journal*, xix., No. 5.

A NOTE ON THE EFFECT OF PRESSURE UPON THE SERIES IN THE SPECTRUM OF AN ELEMENT.

By J. S. AMES AND W. J. HUMPHREYS.

It has been known for many years that the spectra of certain elements, notably the alkalis and the alkaline earths, contained series of lines, which obeyed a mathematical law like that giving the distribution of lines in the spectrum of hydrogen. These series have been thoroughly studied by Kayser and Runge, who have classified them according to their physical characteristics as principal, first subordinate, second subordinate. Only a few elements, lithium, sodium, potassium, have in their spectra all three series; while the last two series, the subordinate ones so-called, are common to some ten others; the wavelengths being different in the spectra of the different elements, the physical properties being the same.

While the shift of the lines in the spectra of the elements was under investigation, it seemed important to study in particular the effect of pressure upon the series of the various elements. To this end, photographs were taken of the arc-spectra of all elements which give series, both at ordinary pressure and at increased pressure; and the shifts were carefully measured of as many of the lines as possible. In certain cases eye-observations were also made. The results for each element may be thus briefly stated:—

1. The lines of any one series of a particular element are shifted alike, *i. e.*, according to the same law, which may be written:—

$$\Delta\lambda = \lambda\beta(p_1 - p_0)$$

where λ is the wave-length, $\Delta\lambda$ is the shift produced by an increase of pressure $p_1 - p_0$, β is a constant for any one series of a definite element.

2. The constant β is different for the different series of the same element, the change being such that, *very nearly*, β for the principal series is one-half β for the first subordinate and one-quarter that of the second subordinate.

3. The constant β is different for the same series of different elements. Special attention is called to this fact in another note in this Circular. One apparent irregularity which demands attention is the fact that, approximately, the value of β for similar elements (*e. g.*, zinc, cadmium, mercury) varies as the cube-root of the atomic weight.

No satisfactory theory has been advanced to account for these shifts of the spectrum lines when the arc is under pressure. There is every evidence that it is not due to a temperature effect of any ordinary kind. It would certainly be expected that the outer envelope of an arc would be at a much lower temperature than the core, and that different electric currents might cause different temperatures in the arc; yet no shift due to these variations has been observed. It is our intention to observe the arc under these conditions with a Michelson refractometer as soon as possible, and so to learn what actually occurs. Again, the temperature of the arc should be much greater than that of a Bunsen flame which is being fed with sodium; but the difference in the wave-length of D_1 under the two conditions is not perceptible with a 21 ft. concave grating, 15,000 lines to the inch; *i. e.*, a shift, if any, must be less than 0.002 of an Angström unit. The words "temperature of the arc" are used with considerable hesitation, because so little is known as to the mean condition of the molecules of the vapour which are producing the light.

One can easily understand, however, that, if the pressure

on a gas is increased, the number of collisions per second must increase; and it is not impossible that this increased internal energy of a molecule, as thus produced, is the immediate cause of a change in the actual size of the molecule. The extent of this change would depend upon the looseness of construction of the molecule, apparently; and this quantity is measured to a certain degree by the coefficient of expansion of the element in the solid form. Therefore, it might be expected that the measured shift would vary in the same direction as the coefficient of expansion of the solid. This is actually the case, with no exception. Again, since on any theory of emission of waves the wave length varies directly as the linear dimension of the portion of matter producing the waves, it would be expected that the measured shift would vary directly as the coefficient of linear expansion, which is found to be the case. If a is this coefficient, it may be stated as an experimental law that, for different elements $\beta = ca$ where c is a constant, which in some way connects the ordinary changes in size of a solid due to temperature changes with the hypothetical changes in the size of the molecules of the vapour due to pressure changes.

It is not difficult to see that one would expect, as consequences of the above ideas, that the shift would be proportional to the total increase of pressure, regardless of its mode of production; and that it should also vary directly as the wave-length in any one series or group of lines, for in such a case the longer waves indicate greater linear dimensions of the vibrating segments, if the term may be used. These are observed phenomena, as is stated above.

The fact that the shift characteristic of a principal series is less than that of the first subordinate, and this in turn less than that of the second subordinate would be expected, in accordance with these ideas, if the molecules producing the principal series were of a simpler structure than those producing the first subsidiary; and if the molecules producing the second subsidiary were the most complex of all. For, since the shift of a series depends upon the looseness of the molecular structure, it would be expected that, if these molecules split up in any way, the fragments would be more stable and firm than the original molecules, and therefore the shift of the original molecules would be greater than that of the fragments. It is difficult, however, to see any reason why the shift of the different series should vary according to any simple law, or why the shift of the same series of different elements should be in accordance with any formula so simple as that of the cube root of the atomic weight. If it could be assumed that the shift was proportional to the linear dimensions of the segment producing the waves, most interesting deductions might be drawn; but there seems to be no justification for the assumption.—*Johns Hopkins University Circular*, xvi., No. 130.

SURFACE TENSION OF WATER AND OF DILUTE AQUEOUS SOLUTIONS.

By N. ERNEST DORSEY.

DURING the past year I have been endeavouring to determine the surface tension of dilute aqueous solutions by means of the method of ripples. All work previously done on the surface tension of solutions has been on solutions of about one-half normal concentration, or greater, and most of the observers have deduced the surface tension from the measured rise of the solution in capillary tubes.

For at least two reasons the method of capillary tubes is open to serious objections. First, the height a liquid rises in a tube depends upon the angle between the wall of the tube and the surface of the liquid where it meets the tube. This contact angle can not possibly be measured, since the surface of the liquid lies entirely on one side of the point where we wish to know its inclination; and as

we can measure the inclination of a finite surface only every measured value of the contact angle must be too large.

The second objection is that probably the surface tension of the solution-glass surface, as well as that of the solution-air surface, varies with the concentration of the solution. If such is the case the surface tension found will depend upon two changes which can not be readily separated, and which render the interpretation of the results difficult.

For these reasons I decided to use the method of ripples, which was first successfully used by Lord Rayleigh, although with his arrangement of apparatus individual observations differ by about 2 per cent. After trying many plans one was finally adopted that gives individual results that agree to about $\frac{1}{4}$ per cent; and the average departure of single observations from the mean of several seldom exceeds $\frac{1}{4}$ per cent.

The waves were generated by a fork whose frequency was often determined and was always near 62.87 double vibrations per second. The water and solutions were contained in a porcelain tray 1 by 12 by 14 inches. The wave length was measured by means of a telescope mounted on a dividing engine, whose screw had a pitch of 1.0328 m.m. The waves were visible under ordinary conditions, but were observed by Foucault's method for rendering visible small vibrations in plane or spherical surfaces.

The water used was especially distilled by Mr. W. T. Mather from chromic acid and alkaline potassium permanganate, and was condensed in a block tin condenser; it was the kind used by him for his electrolytic work. The salts were obtained from Eimer and Amend and were said to be chemically pure.

With this apparatus I have determined the surface tension of water and of solutions of sodium chloride, potassium chloride, sodium carbonate, potassium carbonate, and zinc sulphate, of concentrations varying from 0.05 normal to normal.

The value found for water is $T = 75.98$ dynes per centimetre at 0°C ., while *Sentis Jour. de Phys.* (3) vi., 183, 1897) working by an entirely different method found $T = 76.09$ at 0°C ., which differs from the other by only 0.14 per cent. These values agree very well with the values given by Lord Rayleigh, Hall, Volkmann, and others, but are much lower than Quincke's value.

It was found that the surface tensions of dilute aqueous solutions are linear functions of the concentration; so that we may write $T_8 = T_w + kC$, where T_8 = surface tension of the solution, T_w = surface tension of water at the same temperature, k is a constant, C is the concentration in grm. molecules per litre. Below is a table showing the value of k as determined by different observers.

	Dorsey.	Volkman.	Quincke.	Rother.
NaCl $k =$	1.53	1.59	1.57	1.38
KCl	2.23	1.41	1.57	1.47
$\frac{1}{2}$ Na_2CO_3	2.00	0.987	1.57	—
$\frac{1}{2}$ K_2CO_3	1.77	1.78	1.57	—
ZnSO_4	1.86	—	—	—

Volkmann found that the curve for Na_2CO_3 at great dilution becomes steeper than the one for K_2CO_3 which agrees with my results. Quincke's value, 1.57, does not agree with his results except for KCl and NaCl. I cannot account for the very high value I found for KCl, but it must be borne in mind that the values given above are not fairly comparable, since my values are for solutions generally less concentrated than $\frac{1}{2}$ normal, while the others are found for solutions of greater concentration.—*Johns Hopkins University Circular*, xvi., No. 130.

A Menthoglycol.—Ph. Barbier and G. Lenz.—In addition to isopulegol and menthoglycol, there is formed during the action of dilute sulphuric acid upon citronallal, a small quantity of $\text{C}_{20}\text{H}_{34}\text{O}$, a body boiling at 185° under the pressure of 10 m.m.—*Comptes Rendus*, cxxiv., No. 23.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 23, June 8, 1897.

Correspondence.—The Secretary read a letter announcing the dispatch of 25,000 francs derived from a subscription gathered in Russia as a contribution to the fund for the erection of a monument to Lavoisier.

Properties of Simple Kathodic Rays. Relations with Simple Electric Radiations.—H. Deslandres.—The author's experiments, and in particular an experiment with the Tesla-d'Arsonval arrangement, lead to the following conclusion:—The simple kathodic rays correspond to the simple electric oscillations. Further, the Crookes tube, completed by arrangements described in the author's paper, constitutes an apparatus capable of furnishing rapid and valuable indications on the electrification of conductors submitted to high tensions.

Atomic Weight of Cerium.—M. Wyrouboff and A. Verneuil.—On operating with products strictly pure, and employing methods as free as possible from errors, cerium, of whatever origin, shows an atomic weight very close upon 92.7. Considering the indirect character of the method employed, this figure can only be considered as approximative to about 0.2–0.3.

Heat Liberated on the addition of Bromine to some Non-saturated Substances.—W. Louguine and Jas. Kablukow.—The addition-heats of Br to allylic alcohol and its derivatives vary little among themselves. For allyl chloride and bromide they are almost equal. The substitution of phenyl (C_6H_5) for hydrogen in allylic alcohol diminishes notably the addition-heat of bromine.

Combinations of Phenylhydrazine with Metallic Bromides.—J. Moitessier.—Not suitable for abridgment.

Chemical Study on the Culture of the Cattleya.—Alex Hébert and G. Truffaut.—The generated bulbs contain a diminished proportion of dry matter, of organic and nitrogenous matter, and of ash; the decrease falling principally upon the potassa, lime, magnesia, and phosphoric acid.

Examination of Aluminium Utensils.—M. Balland.—The aluminium utensils must contain 99 to 99.5 of pure aluminium. In alloys of copper and aluminium, the copper must not exceed 2 to 3 per cent. The vessels, &c., must not be cleansed with soda.

Bulletin de la Société Chimique de Paris.

Series 3, Vol. xvii.-xviii., No. 10. May 20, 1897.

Action of Dilute Nitric, Sulphuric, Hydrochloric, and Phosphoric Acids on Nitrates in the Presence of Ether.—C. Tanret.—When two liquids, insoluble in each other, one of which has in solution a body equally soluble in the other, are shaken up together, these two liquids will divide the soluble body between them in such a manner that the quantity as dissolved in given volumes of each will always have a constant relation one to the other. Having occasion to apply this method to the detection of small quantities of nitric acid in a liquid rich in nitrate of ammonia, the author obtained some quite unexpected results. The acid dissolved in ether as an organic acid would have done, then, after agitation with water, passed to a great extent into the latter. Nitric acid therefore possesses two coefficients, according to whether nitrate of ammonia is present or not. This research was the outcome of this observation, and the author maintains that the action of nitrates on the coefficient of "division"

of the nitric acid is explained if we admit that, in dissolving in nitric acid, the neutral nitrate becomes an acid nitrate. Now, water does not completely decompose acid salts into free acid and neutral salts, because these two tend to re-combine and re-form the original salt. It follows, then, that in adding either an excess of acid with regard to the amount of neutral salt present, or an excess of neutral salt with regard to the acid present, the tendency is to augment the stability of the acid salt. This latter case is precisely the result obtained in these experiments.

Formation of Metallic Sulphides by Mechanical Agencies.—L. Franck.—This paper contains an account of several simple experiments, which prove the combination of two bodies by mechanical influence and mutual contact, by the friction of the two bodies. Small quantities of sulphides have been obtained by rubbing flowers of sulphur, and the powder of certain metals, such as iron, copper, aluminium, &c.,—between two sheets of paper.

Action of Chloride of Chloracetyl on some Aromatic Hydrocarbons, in the presence of Chloride of Aluminium.—A. Collet.—In a previous note the author described the action of various chlorides of acid halogens on benzene in the presence of chloride of aluminium; he now describes the products obtained with chloride of chloracetyl in a similar manner.

On some Derivatives of Anise-aldehyd.—A. Reyckler.—Not suitable for abstraction.

Contribution to the Study of Coumarin.—A. Reyckler.—The author finds that by using pure by-products, and working at a sufficiently high temperature, the Perkin reaction does not appear to need the presence of any great proportion of alkaline salts.

Products of the Action of Benzhydrol-diamide and Tetramethyl, on Para- and Meta-sulphanilic Acids.—M. Soais.—Not suitable for abstraction.

On a New Method of Extracting the Perfume from Flowers.—J. Passy.—The author soaks the flowers in water, without killing them; the scent goes into the water, as it does into air; the water is then treated with ether, which collects the scent. This renders maceration unnecessary.

Study of Chlorophyll.—J. Stoklasa.—The author finds that the cellular germ does not form—not only without phosphorus but also without iron—if the chlorophyll contains only phosphorus.

Schiff Reaction applied to Acid Fuschine.—L. Lefevre.—The author finds that M. Cazenueve's conclusion, that fuschine decolourised by SO_2 would not regain its colour when treated with aldehyds, is not correct. The success of the reaction depends entirely on the quantities used.

Bulletin des Travaux de la Société de Pharmacie de Bordeaux. May, 1897.

New Method of Testing for Biliary Calculus.—G. Denigés.—Not suitable for abstraction.

Experiments on Commercial Albumen.—P. Carles.—The adoption of albumen as a clarifier by several industries has brought a number of different brands on the market; some can be used with every confidence, but unfortunately, with a view to cheapen its production, there are also brands carelessly made, and, what is worse, adulterated. Some samples have been found to contain from 12 to 25 per cent of insoluble coagulated matter, having no clarifying power whatever. Gum, dextrine, and gelatin are also used as adulterants. To examine the purity of albumen, 2 grms. should be made into a paste with distilled water, then more water gradually added, up to 200 c.c.; if the albumen be free from coagulated particles this solution will be translucent. To 100 c.c. of

this solution add 35 c.c. of a 1 per cent solution of tannin, then 0.2 grm. of powdered bitartrate of potash. Shake well and filter; to one part of the filtrate add a few drops of a solution of five parts per thousand of grenatine, and to the other a few drops of the tannin solution above mentioned. If there is no change the albumen is pure; if the grenatine gives a precipitate it shows that there is tannin in excess, and, therefore, that the albumen is either adulterated with some inert body or has been over-heated in making; if, however, the tannin gives a precipitate in the last tube, it proves the presence of gelatin in the sample, as gelatin will precipitate, weight for weight, four times as much tannin as dried albumen can.

Phenomenon of Oxidation produced by Different Milks.—R. Dupuy.—Not suitable for abstraction.

New Form of Oven for Desiccation and Sterilisation.—M. Soudard.—The large number of different desiccating ovens now used in laboratories show that perfection is very difficult to attain. Physical science teaches us that the rapidity of desiccation depends on the temperature, on the degree of saturation of the surrounding atmosphere, on the renewing of this atmosphere, and on the amount of surface for evaporation. Most ovens will not allow of the conditions being at their best. The oven made by the author seems to conform to the state of things required. It is made of sheet copper, with double walls, and so ingeniously put together that the inner chamber is heated directly by the flame, while at the same time a current of hot fresh air enters at one side near the bottom, and by means of alternate trays circulates—or rather zigzags—throughout the interior until it reaches the top, where it escapes. The temperature can be so regulated that the oven can be used for evaporations and desiccations, or it can be raised to and maintained at 160° or 200° for sterilisations.

Société de l'Industrie Minérale, Comptes Rendus Mensuels.
April, 1897.

Electrolysis of Solid Bodies.—M. Mayençon.—For these experiments a bichromate battery of six cells is sufficient, and the whole apparatus necessary is of a very simple character—such as an agate mortar, a platinum crucible, one or two spatulas, a glass funnel, &c. The action of the current is shown first on a few simple bodies capable of forming acids; for instance, carbon is transformed by the oxygen of water into carbonic acid, when it is employed as the anode, the cathode being of platinum. To show this a small quantity of baryta water is poured into a test-tube, and a few drops of chloride of barium added; when the current is passed through as described, a white precipitate of carbonate of baryta is formed, soluble in acids. In a like manner sulphur forms sulphuric acid, and then sulphate of barium when moistened with chloride of barium. As a final experiment on insoluble simple bodies, phosphorus was transformed into phosphoric acid, by passing the current through small pieces of phosphorus moistened with nitro-molybdate of ammonia, when the yellow phospho-molybdate of ammonia—insoluble in acids and soluble in ammonia—was formed.

The decomposition of a silicate by means of the electric current is also performed, but it is not easy to show the presence of silica by absolutely characteristic reactions. Orthose gives on one pole silica, at the other potash, and garnierite in a similar manner gives metallic nickel. Further experiments are being made, and will shortly be communicated.

Bulletin de la Société d'Encouragement pour l'Industrie Nationale. Series 5, Vol. ii., No. 5. May, 1897.

Estimation of the Oxidation of Oils.—W. Bishop.—This is a report to the Society by M. Bérard, on the work done by M. Bishop on this subject. This work was inspired by an experiment made by Chevreul in 1856, in which he found that linseed oil mixed with manganese

absorbed ten times more oxygen than the pure oil itself. M. Bishop, however, uses as drying agent a kind of soap made by mixing oxide of manganese with resin. He dissolves this resinate in the oil to be tested in the proportion of 0.2 grm. of the former to 10 grms. of the oil. Then, to facilitate the access of air, he adds 1 grm. of a light silicate, obtained by precipitation and calcination. The desiccation is then allowed to proceed. The table of the order of oxidation confirms what has already been shown by M. Livache with regard to this order. M. Bishop obtained for linseed oil an absorptive value of oxygen of 14 per cent in twenty-four hours. He has successfully applied his method to the comparison of different commercial oils and the recognition of possible mixtures which could be made.

Composition of Clays.—Georges Vogt.—A very long paper, not suitable for abstraction.

Researches on the Colouration of Glass by the Direct Penetration of Metals or Metallic Salts.—Leon Lémal.—Reprinted from the *Comptes Rendus*, May 17, 1897.

On Solutions of Acetylene and their Explosive Properties.—MM. Berthelot and Vieille.—From the *Comptes Rendus*, May 10, 1897.

THE DAVY FARADAY RESEARCH LABORATORY OF THE ROYAL INSTITUTION.

Directors:
The Right Hon. LORD RAYLEIGH, M.A., D.C.L., LL.D., F.R.S.,
Professor DEWAR, M.A., LL.D., F.R.S.

Superintendent of the Laboratory:
Dr. ALEXANDER SCOTT, M.A., D.Sc.

This Laboratory, which has been founded by Dr. LUDWIG MOND, F.R.S., as a Memorial of Davy and Faraday "for the purpose of promoting original research in Pure and Physical Chemistry," is now open. The next Term begins on the 4th of October, 1897.

Under the Deed of Trust, workers in the Laboratory are entitled, free of charge, to Gas, Electricity, and Water, as far as available, and at the discretion of the Directors, to the use of the apparatus belonging to the Laboratory, together with such materials and chemicals as may be authorised.

All persons desiring to be admitted as workers, must send evidence of scientific training, qualification, and previous experience in original research, along with a statement of the nature of the investigation they propose to undertake. Further information, together with forms of application, can be had from the ASSISTANT SECRETARY, Royal Institution.

HULL MUNICIPAL TECHNICAL SCHOOLS. CHEMISTRY MASTERSHIP.

The Technical Instruction Committee is prepared to receive applications for the above Appointment. Candidates must not be under 25 nor over 40 years of age. The Master will not be allowed to undertake any teaching other than that required by the Committee. Salary, £200 per annum, payable monthly. Forms of application and further particulars may be obtained from the undersigned, to whom applications must be sent not later than Wednesday, July 14th, 1897.

J. T. RILEY, D.Sc. (Lond.),
7, Albion Street, Hull. Director of Studies.

THE LEEDS INSTITUTE OF SCIENCE, ART, AND LITERATURE.

The Directors invite applications for the post of HEAD MASTER of The Leeds Technical School and TEACHER of CHEMISTRY to the Boys' and Girls' Modern Schools of the Institute, now vacant by the death of Mr. S. J. Harris, M.Sc.

The Master appointed will be expected to take classes in Theoretical and Practical Inorganic and Organic Chemistry and to exercise a general supervision over the other classes in the Technical School. The school buildings, erected in 1888, are furnished with all necessary materials and apparatus for science teaching.

Salary, partly fixed and partly dependent upon results, amounts to about £325. Full particulars may be had from the SECRETARY, to whom applications must be sent not later than July 20th, 1897.

Canvassing Directors will be considered a disqualification.

THE CHEMICAL NEWS.

VOL. LXXVI., No. 1964.

DIAMONDS.*

By WILLIAM CROOKES, F.R.S., M.R.I.

(Concluded from p. 15).

The Mechanism of the Diamantiferous Pipes.

How the great diamond pipes originally came into existence is not difficult to understand, in the light of the foregoing facts. They certainly were not burst through in the ordinary manner of volcanic eruption; the surrounding and enclosing walls show no signs of igneous action, and are not shattered nor broken even when touching the "blue ground." These pipes after they were pierced were filled from below, and the diamonds formed at some previous epoch too remote to imagine were erupted with a mud volcano, together with all kinds of *débris* eroded from the adjacent rocks. The direction of flow is seen in the upturned edges of some of the strata of shale in the walls, although I was unable at great depths to see any upturning in most parts of the walls of the De Beers mine.

Let me again refer you to the picture of the section through the Kimberley mine. There are many such pipes in the immediate neighbourhood. It may be that each volcanic pipe is the vent for its own special laboratory—a laboratory buried at vastly greater depths than we have reached or are likely to reach—where the temperature is comparable with that of the electric furnace, where the pressure is fiercer than in our puny laboratories and the melting-point higher, where no oxygen is present, and where masses of carbon-saturated iron have taken centuries, perhaps thousands of years, to cool to the solidifying point. Such being the conditions, the wonder is, not that diamonds are found as big as one's fist, but that they are not found as big as one's head. The chemist arduously manufactures infinitesimal diamonds, valueless as ornamental gems; but Nature, with unlimited temperature, inconceivable pressure, and gigantic material, to say nothing of measureless time, produces without stint the dazzling, radiant, beautiful crystals I am enabled to show you to-night.

The ferric origin of the diamond is corroborated in many ways. The country round Kimberley is remarkable for its ferruginous character, and iron-saturated soil is popularly regarded as one of the indications of the near presence of diamonds. Certain artificial diamonds present the appearance of an elongated drop. From Kimberley I have with me diamonds which have exactly the appearance of drops of liquid separated in a pasty condition and crystallised on cooling. At Kimberley and in other parts of the world, diamonds have been found with little appearance of crystallisation but with rounded forms similar to those which a liquid might assume if kept in the midst of another liquid with which it would not mix. Other drops of liquid carbon retained above their melting-point for sufficient time would coalesce with adjacent drops, and on slow cooling would separate in the form of large perfect crystals. Two drops, joining after incipient crystallisation, would assume the not uncommon form of interpenetrating twin crystals. Illustrations of these forms from Kimberley are here to-night. Other modified circumstances would produce diamonds presenting a confused mass of boarty crystals, rounded and amorphous masses, or a hard black form of carbonado.

Again, diamond crystals are almost invariably perfect

on all sides. They show no irregular side or face by which they were attached to a support, as do artificial crystals of chemical salts; another proof that the diamond must have crystallised from a dense liquid.

When raised the diamond is in a state of enormous strain, as I have already shown by means of polarised light. Some diamonds exhibit cavities which the same test proves to contain gas at considerable pressure.

The ash left after burning a diamond invariably contains iron as its chief constituent; and the most common colours of diamonds, when not perfectly pellucid, show various shades of brown and yellow, from the palest "off colour" to almost black. These variations accord with the theory that the diamond has separated from molten iron, and also explains how it happens that stones from different mines, and even from different parts of the same mine, differ from each other. Along with carbon, molten iron dissolves other bodies which possess tinctorial powers. One batch of iron might contain an impurity colouring the stones blue, another lot would tend towards the formation of pink stones, another of green, and so on. Traces of cobalt, nickel, chromium, and manganese, metals present in the blue ground, might produce all these colours.

An hypothesis, however, is of little value if it only elucidates one half of a problem. Let us see how far we can follow out the ferric hypothesis to explain the volcanic pipes. In the first place we must remember these so-called volcanic vents are admittedly not filled with the eruptive rocks, scoriaceous fragments, &c., constituting the ordinary contents of volcanic ducts. At Kimberley the pipes are filled with a geological plum pudding of heterogeneous character—agreeing, however, in one particular. The appearance of shale and fragments of other rocks shows that the *mélange* has suffered no great heat in its present condition, and that it has been erupted from great depths by the agency of water vapour or some similar gas. How is this to be accounted for?

It must be borne in mind I start with the reasonable supposition that at a sufficient depth* there were masses of molten iron at great pressure and high temperature, holding carbon in solution, ready to crystallise out on cooling. In illustration I may cite the masses of erupted iron in Greenland. Far back in time the cooling from above caused cracks in superjacent strata through which water† found its way. On reaching the iron the water would be converted into gas, and this gas would rapidly disintegrate and erode the channels through which it passed, grooving a passage more and more vertical in the endeavour to find the quickest vent to the surface. But steam in the presence of molten or even red-hot iron rapidly attacks it, oxidises the metal and liberates large volumes of hydrogen gas, together with less quantities of hydrocarbons† of all kinds—liquid, gaseous, and solid. Erosion commenced by steam would be continued by the other gases, and it would be no difficult task for pipes, large as any found in South Africa, to be scored out in this manner. Sir Andrew Noble has shown that when the screw stopper of his steel cylinders in which gunpowder explodes under pressure is not absolutely perfect, gas finds its way out with a rush so overpowering as to score a wide channel in the metal; some of these stoppers and vents are on the table. To illustrate my argument Sir Andrew Noble has been kind enough to try a special experiment. Through a cylinder of granite is drilled a hole 0.2 inch diameter, the size of a small vent. This is made the stopper of an explosion chamber, in which a quantity of cordite is fired, the gases escaping through the granite vent. The pressure is about 1500 atmospheres, and the whole time of escape

* The requisite pressure of fifteen tons on the square inch would exist not many miles beneath the surface of the earth.

† There are abundant signs that a considerable portion of this part of Africa was once under water, and a fresh-water shell has been found in apparently undisturbed blue ground at Kimberley.

‡ The water sunk in wells close to the Kimberley mine is sometimes impregnated with paraffin, and Sir H. Roscoe extracted a solid hydrocarbon from the "blue ground."

* A Lecture delivered at the Royal Institution, Friday, June 11th, 1897.

is less than half a second. Notice the erosion produced by the escaping gases and by the heat of friction, which have scored out a channel over half an inch diameter and melted the granite along their course. If steel and granite are thus vulnerable at comparatively moderate gaseous pressure, is it not easy to imagine the destructive upburst of hydrogen and water-gas grooving for itself a channel in the diabase and quartzite, tearing fragments from resisting rocks, covering the country with débris, and finally, at the subsidence of the great rush, filling the self-made pipe with a water-borne magma in which rocks, minerals, iron oxide, shale, petroleum, and diamonds are churned together in a veritable witch's cauldron! As the heat abated the water vapour would gradually give place to hot water, which forced through the magma would change some of the mineral fragments into the now existing forms.

Each outbreak would form a dome-shaped hill, but the eroding agency of water and ice would plane these eminences until all traces of the original pipes were lost.

Actions, such as I have described, need not have taken place simultaneously. As there must have been many molten masses of iron with variable contents of carbon, different kinds of colouring matter, solidifying with varying degrees of rapidity, and coming in contact with water at intervals throughout long periods of geological time—so must there have been many outbursts and upheavals, giving rise to pipes containing diamonds. And these diamonds, by sparseness of distribution, crystalline character, difference of tint, purity of colour, varying hardness, brittleness and state of tension, would have impressed upon them, engraved by natural forces, the story of their origin—a story which future generations of scientific men may be able to interpret with greater precision than we can to-day.

Who knows but that at unknown depths in the earth's metallic core beneath the present pipes there are still masses of iron not yet disintegrated and oxidised by aqueous vapour,—masses containing diamonds, unbroken, and in greater profusion than they exist in the present blue ground, inasmuch as they are enclosed in the matrix itself, undiluted by the numerous rock constituents which compose the bulk of the blue ground.

Meteoritic Diamonds.

There is another diamond theory which appeals to the fancy. It is said that the diamond is a direct gift from Heaven, conveyed to earth in meteoric showers. The suggestion, I believe, was first broached by A. Meydenbauer (*CHEMICAL NEWS*, lxi., p. 209, 1890), who says:—"The diamond can only be of cosmic origin, having fallen as a meteorite at later periods of the earth's formation. The available localities of the diamond contain the residues of not very compact meteoric masses, which may, perhaps, have fallen in historic ages, and which have penetrated more or less deeply, according to the more or less resistant character of the surface where they fell. Their remains are crumbling away on exposure to the air and sun, and the rain has long ago washed away all prominent masses. The enclosed diamonds have remained scattered in the river beds, while the fine light matrix has been swept away."

According to this hypothesis, the so-called volcanic pipes are simply holes bored in the solid earth by the impact of monstrous meteors—the larger masses boring the holes, while the smaller masses, disintegrating in their fall, distributed diamonds broadcast. Bizarre as such a theory may appear, I am bound to say there are many circumstances which show that the notion of the heavens raining diamonds is not impossible.

In 1846 a meteorite fell in Hungary (the "Ava meteorite") which was found to contain graphite in the cubic crystalline system. G. Rose thought this cubic graphite was produced by the transformation of a diamond. Long after this prediction was verified by Weinschenk, who found transparent crystals in the Ava meteorite. Mr. Fletcher has found in two meteoric irons

—one from Yondeggin, East Australia, and one from Crosby's Creek, United States—crystals absolutely similar to those in the Ava meteorite.

In 1886 a meteorite falling in Russia contained, besides other constituents, about 1 per cent of carbon in light grey grains, having the hardness of diamond, and burning in oxygen to carbonic acid.

Daubrée says the resemblance is manifest between the diamantiferous earth of South Africa and the Ava meteorite, of which the stony substance consists almost entirely of peridot. Peridot being the inseparable companion of meteoric iron, the presence of diamonds in the meteorites of Ava, of Yondeggin, and of Crosby's Creek, bring them close to the terrestrial diamantiferous rocks.

Hudleston maintains that the bronzite of the Kimberley blue ground is in a condition much resembling the bronzite grains of meteorites; whilst Maskelyne says that the bronzite crystals of Dutoitspan resemble closely those of the bronzite of the meteor of Breitenbach, but are less rich in crystallographic planes.

But the most striking confirmation of the meteoric theory comes from Arizona. Here, on a broad open plain, over an area about five miles diameter, were scattered one or two thousand masses of metallic iron, the fragments varying in weight from half a ton to a fraction of an ounce. There is little doubt these masses formed part of a meteoric shower, although no record exists as to when the fall took place. Curiously enough, near the centre, where most of the meteorites have been found, is a crater with raised edges, three-quarters of a mile in diameter, and about 600 feet deep, bearing exactly the appearance which would be produced had a mighty mass of iron or falling star struck the ground, scattered in all directions, and buried itself deep under the surface. Altogether ten tons of this iron have already been collected, and specimens of the Canyon Diablo meteorite are in most collectors' cabinets.

An ardent mineralogist, the late Dr. Foote, in cutting a section of this meteorite, found the tools were injured by something vastly harder than metallic iron, and an emery wheel used in grinding the iron had been ruined. He examined the specimen chemically, and soon after announced to the scientific world that the Canyon Diablo meteorite contained black and transparent diamonds. This startling discovery was afterwards verified by Professors Friedel and Moissan, who found that the Canyon Diablo meteorite contained the three varieties of carbon—diamond (transparent and black), graphite, and amorphous carbon. Since this revelation, the search for diamonds in meteorites has occupied the attention of chemists all over the world.

I am enabled to show you photographs of true diamonds I myself have extracted from pieces of the Canyon Diablo meteorite, five pounds of which I have dissolved in acids for this purpose—an act of vandalism in the cause of science for which I hope mineralogists will forgive me. A very fine slab of the meteorite, weighing about seven pounds, which has escaped the solvent, is on the table before you.

Here, then, we have absolute proof of the truth of the meteoric theory. Under atmospheric influences the iron would rapidly oxidise and rust away, colouring the adjacent soil with red oxide of iron. The meteoric diamonds would be unaffected, and would be left on the surface of the soil to be found by explorers when oxidation had removed the last proof of their celestial origin. That there are still lumps of iron left at Arizona is merely due to the extreme dryness of the climate and the comparatively short time that the iron has been on our planet. We are here witnesses to the course of an event which may have happened in geologic times anywhere on the earth's surface.

Although in Arizona diamonds have fallen from above, confounding all our usual notions, this descent of precious stones seems what is called a freak of Nature rather than a normal occurrence. To the modern student of science

there is no great difference between the composition of our earth and that of extra-terrestrial masses. The mineral peridot is a constant extra-terrestrial visitor, present in most meteorites. And yet no one doubts that peridot is also a true constituent of rocks formed on this earth. The spectroscope reveals that the elementary composition of the stars and the earth are pretty much the same; so does the examination of meteorites. Indeed, not only are the selfsame elements present in meteorites, but they are combined in the same way to form the same minerals as in the crust of the earth.

This identity between terrestrial and extra-terrestrial rocks recalls the masses of nickeliferous iron of Ovifak. Accompanied with graphite they form part of the colossal eruptions which have covered a portion of Greenland. They are so like meteorites that at first they were considered to be meteorites till their terrestrial origin was proved. They contain as much as 1·1 per cent of free carbon.

It is certain from observations I made at Kimberley, corroborated by the experience gained in the laboratory, that iron at a high temperature and under great pressure will act as the long-sought solvent for carbon, and will allow it to crystallise out in the form of diamond—conditions existent at great depths below the surface of the earth. But it is also certain, from the evidence afforded by the Arizona and other meteorites, that similar conditions have likewise existed among bodies in space, and that a meteorite freighted with its rich contents, on more than one occasion has fallen as a star from the sky. In short, in a physical sense, Heaven is but another name for Earth, or Earth for Heaven.

A NEW FORM OF REFLECTING TELESCOPE.

By CHARLES LANE POOR.

DR. POOR explained some experiments that he has carried out in grinding and polishing a new form of parabolic mirror for reflecting telescopes. The mirror is a portion of a paraboloid of revolution cut at the extremity of the latus rectum. The reflected beam is at right-angles to the incident light; no second mirror is therefore necessary; the full aperture of the mirror being used.

The advantages of such a form of mirror were pointed out, and the great simplification in equatorial mountings indicated. The declination axis becomes the telescope tube, the image being formed at the intersection of the polar and declination axis and is always in the same position; the observer remains at rest while viewing any and every part of the visible heavens. No dome is required. Other advantages were indicated, and several modifications of the general form pointed out—*Johns Hopkins University Circular*, xvi., No. 130.

TESTIMONIAL TO PROF. ATTFIELD, F.R.S.

AN interesting ceremony was performed on Saturday afternoon last, the 10th July, when Prof. Attfield was presented with a testimonial on the occasion of his retirement from the Chair of Practical Chemistry in the School of the Pharmaceutical Society of Great Britain, after having occupied the position for thirty-four years.

The testimonial took the form of an album containing the signatures of one thousand old pupils and two hundred other friends of Prof. Attfield, a silver tray, and a silver tea and coffee service—all of which were suitably inscribed.

The presentation was made at Prof. Attfield's residence, at Ashlands, Watford, where Mrs. Attfield had a garden-party and reception. We trust he will live long to enjoy the rest he has so well earned.

HYPOIODOUS ACID AND HYPOIODITES.*

By R. L. TAYLOR, F.C.S.

(Concluded from p. 20).

Hypoiodous Acid.

So far as I have been able to ascertain, it has always been stated that all attempts to obtain hypoiodous acid by the action of iodine and water upon mercuric oxide have failed, and that nothing but iodic acid was formed.† I had tried the action once more with the aqueous solution of iodine, and had apparently been as unsuccessful as ever. I had succeeded, as I believe, in obtaining the acid by other methods, to be presently described, and had noticed the curious anomaly that the free acid, or what I took to be the free acid, bleached indigo with far less energy than the hypoiodites described in the first part of this paper; but I had failed, as others no doubt had frequently failed, to obtain any bleaching solution by the action of iodine and water upon mercuric oxide.

In the paper already referred to by Walker and Kay, the authors state that they "made a solution of hypoiodous acid by agitating pure aqueous solution of iodine with mercuric oxide," filtered, neutralised with potash, and added magnesium sulphate, so obtaining a white precipitate. The addition now of a few drops of potassium iodide *stained the precipitate brown*. This certainly pointed to the presence of a hypoiodite, which would be decomposed, with liberation of iodine, by potassium iodide. I thought the reaction might *possibly* be due to iodic acid, which is usually said to be the sole product of the action of iodine on mercuric oxide; but experiment convinced me that it was not. Further investigation soon showed that hypoiodous acid is really produced when iodine water is shaken up with mercuric oxide (the precipitated oxide is far the best) and filtered. Many others besides Walker and Kay have no doubt prepared the acid in this way, but failed to recognise it. The explanation is that the bleaching action of the free acid is *excessively feeble* as compared with Schönbein's solutions. Contact with the indigo solution for a long time causes the colour slowly to disappear, *but the addition of a drop of alkali immediately transforms the acid into as strong a bleaching solution as Schönbein's solutions*.

I may at once state what appears to me a possible explanation of what seems at first sight a most extraordinary anomaly—that a free acid has a very much feebler oxidising power than one of its salts! When hypoiodous acid bleaches, I suppose it does so according to the following equation:— $\text{HOI} = \text{HI} + \text{O}$. It thus produces hydriodic acid, an extremely unstable body itself, and, further, a compound which would immediately decompose the remaining hypoiodous acid. On the other hand, in presence of an alkali, the result of losing oxygen would be to produce sodium or potassium iodide, both perfectly stable bodies, and with any tendency to decom-

* From *Memoirs and Proceedings of the Manchester Literary and Philosophical Society*, vol. xli., Part III.

† So long ago as 1845, Köne (*Poggendorff's Ann.*, lxvii., p. 302) tried the experiment of shaking up precipitated mercuric oxide with an alcoholic solution of iodine, and, from the fact that he obtained an unstable solution which gradually liberated iodine, and from analogy with the chlorine compounds, he concluded that hypoiodous acid was formed. I have repeated the experiment, and there certainly appears to be a hypoiodous compound produced; the alcohol which is present, however, interferes with the reactions. The filtered liquid contains a considerable amount of mercury. It gives a yellowish precipitate with water, and if a little alkali is added to this it possesses very strong bleaching properties. The precipitate produced with water dissolves up in sodium hydrate, but in a few seconds another precipitate appears which is manifestly iodoform; at the same time the liquid loses its bleaching power. This appears to point to the conclusion that the formation of a hypoiodide is the necessary prelude to the formation of iodoform (see Van Deventer and van't Hoff, *Chem. Central.*, 1888, p. 362). On account of this rapid change in presence of an alkali, the solution does not give the cobalt reaction. If the precipitate produced by water in the alcoholic solution is allowed to stand for some hours, scarlet mercuric iodide separates out. The alcoholic solution is moderately stable, but it gradually liberates iodine on standing.

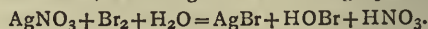
pose the remaining hypoiodite counteracted by the presence of the alkali. Then there is also the further consideration that apparently hypoiodous acid can only have about one-half the total bleaching power that a hypoiodite will have, because, as soon as any of it does bleach it produces hydriodic acid, which would immediately decompose an equivalent amount of the remaining hypoiodous acid. (In one comparative experiment that I made, the bleaching power of the free acid was almost exactly half that of an equal volume of the same iodine solution to which soda had been added. But the bleaching continued for *two hours*, so that the effect would be complicated by the spontaneous decomposition of the free acid).

When a little alkali is added to the hypoiodous acid prepared in this way, the solution behaves almost exactly like Schönbein's solutions. It bleaches strongly, and some determinations I have made with the standard indigo solution gave a bleaching action equivalent to 80 per cent of the iodine used,—that is, representing 40 out of a possible 50 per cent of iodine existing as hypoiodous acid. This result again is confirmed by experiments by Schwicker's method, only in the case of this solution, when it is neutralised by an alkali, it forms nothing else but hypoiodite, and consequently the addition of soda-water simply liberates hypoiodous acid, and there is no separation of iodine. In order to complete the determination potassium iodide has to be added, when there is an immediate liberation of iodine. A determination by this method, which is probably more accurate than the bleaching method, gave liberated iodine equal to 90 per cent of that originally used, so that 45 out of a possible 50 per cent of iodine existed in the solution as hypoiodous acid.

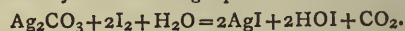
The hypoiodous acid solution to which a little alkali has been added gives a precipitate with cobalt solution, which gradually turns brown and then black, and an immediate brown precipitate with a manganous salt. With silver nitrate it gives a buff-colored precipitate similar to the one I had previously obtained from Schönbein's solutions, but in this case of course it will not be mixed with so much silver iodide. If nitrate of silver is added to the solution containing the free acid a milkiness is produced, and on boiling there is a further precipitate, which contains silver iodide and iodate; the iodate can be dissolved out by ammonia. This reaction I had previously noticed with the acid prepared in another way. The hypoiodous acid is manifestly converted into hydriodic and iodic acids, $3\text{HIO} = 2\text{HI} + \text{HIO}_3$. The aqueous solution of the free acid appears to be moderately stable. When it decomposes, as one would anticipate, it appears to do so according to the equation given above, only in this case the two acids immediately decompose each other, liberating iodine.* In the case of the solution made, as described, with the use of aqueous solution of iodine, the appearance of free iodine in the liquid is very slow; but, by using iodine-water with a little suspended precipitated iodine, a much stronger solution of the hypoiodous acid is obtained, in which the brown colour of iodine begins to show itself within a minute or two of its being filtered. (It appears also that some mercury comes through, because on standing for some hours there is a slight deposit of the scarlet iodide of mercury). After standing for some time, this solution, which manifestly contains free iodine (carbon bisulphide shaken up with it is coloured deep violet), gives no blue colour with starch until the addition of a drop of alkali, or until it has been exposed to the air for some time. If a little of the mixture of the brown solution with starch is poured into a shallow

vessel, or is poured backwards and forwards from one vessel to another, it soon turns blue.*

Some time before obtaining the free hypoiodous acid in the way described above, I had obtained what must have been either the acid or a solution of its silver salt by another method. It was pointed out by Dancer (*Chem. Soc. Journ.*, xv., 447) that hypobromous acid could be obtained by the action of bromine-water upon solution of nitrate of silver, according to the following equation:—



Chlorine acts in a similar way, producing hypochlorous acid. A solution of nitrate of silver with powdered iodine appears to form nothing but iodide and iodate of silver; but I found that iodine suspended in water gave a bleaching liquid when shaken up with solution of silver sulphate, or with a paste of silver carbonate, the carbonate giving the better result. Wishing to obtain some quantitative results, I began to use an aqueous solution of iodine, instead of having it merely suspended in water. Shaking up this solution with a little silver carbonate and rapidly filtering, a liquid is obtained which contains a small amount of silver, but which gives all the reactions which I have described as characteristic of Schönbein's solutions, complicated a little, in some cases, by the silver which is present. It bleaches indigo slowly, but much more rapidly than the acid prepared with mercuric oxide; it also bleaches cochineal and logwood, and oxidises cobalt and manganese salts in presence of an alkali. The action of the iodine upon the silver carbonate may probably be represented by the following equation:—



If a drop of silver nitrate is added to the solution and the liquid boiled, a yellow precipitate is produced, containing iodide and iodate of silver.

I made many attempts to ascertain, by various methods, the proportion of the iodine used which was converted into hypoiodous acid. I filtered the liquid into an acidified solution of potassium iodide, whereby iodine is liberated, the amount of which I estimated. Another method tried was to allow the liquid to run into a standard solution of sulphurous acid, and then to find the amount of this which was oxidised. The results were not satisfactory, as I seldom found that more than 50 per cent of the iodine was converted into bleaching iodine. I have reason to believe that this result was due to some insoluble hypoiodite of silver being left in the precipitate. Finally I tried the standard indigo solution, and found that it was possible to estimate the bleaching power even in the muddy liquid containing the excess of silver carbonate and the silver iodide which was produced in the reaction. Shaking up a known volume of the iodine solution with silver carbonate and immediately running in the indigo solution, the bleaching action indicated about 50 to 60 per cent of the theoretical amount; but the addition of a few drops of dilute sulphuric acid carried the bleaching power still further, owing no doubt to the decomposition of some insoluble silver hypoiodite by the acid. By adding the dilute acid immediately after shaking together the iodine and the silver carbonate, a bleaching action was obtained equal to from 90 to 95 per cent of the theoretical amount. Subsequently I found that, with the aqueous solution of iodine, nitrate of silver reacts perfectly well, producing a bleaching liquid which gives all the reactions already described, complicated a little by the presence of excess of silver. The bleaching action, immediately after the silver nitrate has been added, indicates 95 per cent of the theoretical amount.

The solutions prepared by the use of these silver salts.

* It will be somewhat difficult to measure the rate at which the hypoiodous acid decomposes. Neither of the two methods mentioned in the text for estimating the amount of hypoiodite is applicable when once the decomposition of the acid has started, because each of them would require the preliminary addition of a little alkali, and this would at once convert any liberated iodine into iodide and hypoiodite.

* Dr. A. Harden tells me that he has found that a mixture of aqueous iodine and iodic acid, in certain proportions, behaves in exactly the same way as the above solution. It is known that iodine forms no blue compound with starch unless an iodide be present; but I can offer no explanation of the effect which the air appears to have on the above reaction.

are extremely unstable; that made with the silver nitrate loses 90 per cent of its bleaching power on standing five minutes. This I think may be attributed to the presence of silver in the solution, and to the tendency of the iodine and silver to form the insoluble silver iodide. The solutions prepared in this way are considerably more sluggish in their bleaching action than the alkaline hypiodites, but very much more rapid than the free acid prepared by the mercuric oxide method. If the explanation which I suggested for the difference between the free acid and Schönbein's solutions is correct, it seems to me that it would apply in this case as well. There is silver present in the solutions, and therefore when the bleaching is finished the final product will be silver iodide, a much more stable body than hydriodic acid.

I have to thank Mr. G. P. Varley, B.Sc., and Mr. J. H. Wolfenden, B.Sc., for assistance given me in some portions of this work.

ON THE VOLUMETRIC DETERMINATION OF ZINC BY POTASSIUM FERROCYANIDE.

By L. L. DE KONINCK and EUG. PROST.

(Continued from p. 26).

E.—THE zincic solution is placed in a matrass graduated to 200 c.c.; we add 25 c.c. of ferrocyanide, and fill with water up to the mark, shake well, then let stand. The precipitate behaves as in experiments A and B. After standing three hours, we take 100 c.c. of the clear liquid and titrate directly, in the cold, with ferrocyanide solution, until the mixture when tested with uranium gives a very faint brown tint, even if the test is repeated after a few minutes. Using 11.30 c.c. with an extra two drops of ferrocyanide, the uranium test gives a distinct colouration, showing an excess of the reagent. Total ferrocyanide used for 25 c.c. of zincic solution:—

$$25 + (2 \times 11.30) = 47.60,$$

instead of 50 c.c., the theoretical quantity. The importance of this test, from the point of view of obtaining an exact knowledge of the reaction, decided us to repeat it under slightly different conditions.

E'. We used the same zincic solution, $\frac{1}{2}$ normal, but a solution of ferrocyanide, $\frac{1}{2}$ normal for zinc,—that is to say, corresponding exactly to that of zincic chloride under the conditions of the formation of $K_2Zn_3Fe_2Cy_{12}$: 25 c.c. of the zincic chloride, diluted with 100 c.c. of water acidulated with hydrochloric acid, are placed in a flask graduated to 200 c.c.; we run in 13 c.c. of ferrocyanide, and fill with water up to the mark. The precipitate is very gelatinous, and remains so. After standing forty-eight hours it has not changed, and still occupies two-thirds of the total volume; the supernatant liquid is perfectly clear.

Filter off 100 c.c. of clear liquid (I.). That part of the precipitate remaining on the filter is completely removed; the filter is washed, and the washings added to the part remaining in the flask (II.) This experiment was done in duplicate.

I. To determine the quantity of zinc remaining in (I.) we added 6 c.c. of ferrocyanide. After digesting for fifteen minutes the precipitate becomes of an opaque white colour; we found with nitrate of uranium the presence of an excess of the reagent, which is titrated back with chloride of zinc.

	First test.	Second test.
$ZnCl_2$ — $\frac{1}{2}$ normal used ..	1.70 c.c.	1.75 c.c.

The end of the reaction is shown very sharply.

II. To the troubled liquor (II.) we first add, as a preliminary, 6 c.c. of ferrocyanide; an immediate uranium test gives an intense reaction, but after a few instants no

reaction at all. Add a further 2 c.c. of ferrocyanide; an immediate test again gives a very marked reaction, but after a few seconds only a faint tint, until, after a further addition of 1 c.c., the test shows an excess of the reagent even after standing for some minutes.

In the second parallel operation we used at once 9 c.c. of ferrocyanide. On the precipitate becoming white, we titrated back with zincic chloride after a lapse of twenty minutes,

	First test.	Second test.
$ZnCl_2$ — $\frac{1}{2}$ normal used ..	1.25 c.c.	1.35 c.c.

The end of the reactions was not so distinctly marked; this we attribute to the relatively large quantity of the precipitate.

The conclusion to be drawn from the experiments E and E' is that, even in the presence of a large excess of zincic salt (50 and 48 per cent), the precipitate produced by the ferrocyanide is formed principally of zincicopotassic ferrocyanide, but contains nevertheless zincic ferrocyanide; this latter reacts with the potassic salt, and combines with it so as to form $K_2Zn_3Fe_2Cy_{12}$.

If the ferrocyanide is in sufficient proportion, or, better still, in excess, the precipitate is formed exclusively of the compound $K_2Zn_3Fe_2Cy_{12}$. At the moment of its formation this body is gelatinous, and reacts sharply with salts of uranium; but it aggregates rapidly, and soon gives no further such reaction.

Formed in presence of an excess of zinc, the precipitate contains a small proportion of zincic ferrocyanide; that is, at least, what appears to result from the last experiment, since titration only detected respectively 90.4, 71.7, and 70.8 per cent of the quantities which should be present if the precipitate had been formed solely of $K_2Zn_3Fe_2Cy_{12}$.

But whatever may be the form under which this excess of zinc exists in the first precipitate, it still reacts with the ferrocyanide in such a manner as to finally produce $K_2Zn_3Fe_2Cy_{12}$, but without doubt more slowly than if it were in solution.

IV. Research on the best Method of Working to be used.

It would appear, from what has preceded, that the reaction would be more regular, and its termination more sharply marked, if we proceeded in such a manner that the ferrocyanide should be always, as far as possible, in excess with regard to the zinc,—that is to say, if we added the zincic solution to the ferrocyanide, instead of running this latter into that containing the zinc; this has already been noted by Zulkowski. To effect this it is necessary, in estimating zinc, either to operate, by inverse titration, which is not very practicable, or else to titrate back. This method of working has, further, an advantage observed by Mohr (*Mohr-Classen*, 1886, p. 458) of letting us know when we are approaching the end of the reaction, this being shown by the diminution of intensity of the colouration produced by the indicator, while in direct titration the termination is shown suddenly, and it is easy to overshoot the mark.

For these reasons we have given the preference to titrating back.

We would wish, however, to mention what accuracy can be obtained by direct titration. Our experiments in this direction having soon brought us to the conclusion that titrating back is much more preferable, we will not go into much detail, but will content ourselves with saying that working in the cold, either in hydrochloric or acetic solution, the end of the reaction is very indistinct; thus, as we have already shown, the drop test, done immediately after the addition of the ferrocyanide, gives a distinct colouration, diminishing in clearness as we leave a longer time between the addition of the liquid and taking the drop. By stopping the estimation at the moment when the nitrate of uranium begins to show, we, as a rule, obtain results which cannot be called very exact, varying considerably with the conditions of the experi-

ment,—that is to say, with the state of the precipitate; if the operation is carried out warm, the reaction is sharper. By working slowly, under definite conditions, especially using heat, we can, however, obtain results which might be considered satisfactory if we did not possess, in "titrating back," a much more satisfactory method.

Titration back with permanganate of potash has already been proposed by Renard (*Comptes Rendus*, vol. lxvii., p. 450, 1868), but this method necessitates the removal of the precipitate by filtration, and the zincic or zincicopotassic ferrocyanide reacts with the permanganate. The back titration by means of a solution of zincic chloride enabled us to obtain very satisfactory results, either with solutions of salts of pure zinc or with minerals. It now remains to establish by experiment the most favourable conditions of working.

Influence of Time.—From what we have seen, a great deal depends on whether we perform the "touch" test with nitrate of uranium, immediately after the mixture of the zincic salt and the ferrocyanide, or whether we wait for a few minutes. It was therefore necessary to determine the minimum time, after which the test gives constant results.

In the following experiments we proceeded each time by pouring 20 c.c. ZnCl_2 , $\frac{1}{2}$ normal, into a mixture containing—

50 c.c. ferrocyanide, $\frac{1}{2}$ normal for zinc;
10 c.c. HCl , 5 normal;
50 c.c. AmCl , at 20 per cent; *
100 c.c. water.

Back titration by the same $\frac{1}{2}$ normal solution of zincic chloride:—

1. Titration done as rapidly as possible, used for this 5'50 ZnCl_2 ; total, 25'50.
2. Titration done at once, but without undue haste, used 5'00 ZnCl_2 ; total 25'00.
3. Titration after 7'5 minutes, used 4'65 ZnCl_2 ; total, 24'65.
4. Titration after 15 minutes, used 4'55 ZnCl_2 ; total, 24'55.
5. Titration after 30 minutes, used 4'55 ZnCl_2 ; total, 24'55.

Fifteen minutes therefore suffice to obtain a complete transformation of the precipitate.

Influence of the Initial Excess of Ferrocyanide.—We may ask if a notable excess of potassic ferrocyanide is necessary to transform, integrally and rapidly, into a double cyanide the small quantity of zincic ferrocyanide which seems to form when we mix the alkaline ferrocyanide and the zinc solution. The following series of experiments answer this question:—Five solutions were prepared, each containing, as in the preceding series, 10 c.c. HCl , 5 normal; 50 c.c. AmCl at 20 per cent, and 100 c.c. of water; then respectively 5, 10, 15, 20, and 24 c.c. of ZnCl_2 , $\frac{1}{2}$ normal. Into each was run 50 c.c. of $\frac{1}{2}$ normal ferrocyanide, and titrated back after a quarter of an hour's digestion. This required respectively—

19'50, 14'55, 9'50, 4'45, 0'80 ZnCl_2 ,

or a total, for the 50 c.c. of ferrocyanide, of—

24'50, 24'55, 24'50, 24'45, 24'80 ZnCl_2 .

We see from these results that an excess corresponding to 5 c.c. on 25, or 20 per cent is quite sufficient to produce the transformation in fifteen minutes. The only result which is notably away from the average is that where the excess of ferrocyanide corresponds to less than 1 c. of the zincic solution.

Influence of the Order of Mixing.—It is indifferent whether the zinc solution is poured into the ferrocyanide

or *vice versa*. We have in many assays, worked either one way or the other, without noticing the slightest difference in the results, or even in the rapidity with which the molecular transformation of the precipitate takes place. Special tests have moreover proved it.

Influence of Chloride of Ammonium.—In each of the experiments in the two following series we used 20 c.c. ZnCl_2 , $\frac{1}{2}$ normal; 50 c.c. of ferrocyanide, $\frac{1}{2}$ normal; 10 c.c. of hydrochloric acid, 5 normal; and 150 c.c. of a mixture of water and a 20 per cent solution of ammonium chloride, in the proportions given below, titrating back with the same zincic solution.

AmCl.	H_2O .	ZnCl_2 .		ZnCl_2 Total.	
		Series I.	Series II.	Series I.	Ser. II.
1.	0	150	5'05	—	—
2.	—	—	5'00	4'80	24'80
3.	25	125	4'75	4'40	24'75
4.	50	100	4'55	4'33	24'55
5.	75	75	4'60	4'24	24'60
6.	100	50	4'50	4'10	24'50
7.	125	25	4'55	4'05	24'55
8.	150	0	4'75	3'85	24'75

(To be continued).

PRECIPITATION OF COPPER BY MAGNESIUM.

By E. G. BRYANT.

I was induced, by a paragraph in the South Kensington Chemistry Syllabus, to try some experiments on the action of magnesium on solutions of copper salts, and though the method used was necessarily extremely crude, the results seemed so decisive that I thought they might be of interest to others. It was intended that the work, if successful, should be attempted by mere beginners in practical chemistry; therefore the method used was made as simple as possible, all filtering being dispensed with. The materials used were commercial magnesium ribbon, copper sulphate, and chloride. The magnesium was thoroughly cleaned and the CuSO_4 re-crystallised. After precipitation, the copper was well washed by decantation, dried, and weighed either as copper or copper oxide. My experience seems to show that it is easier, unless a furnace or foot blowpipe be at hand, to get good results with metallic copper than with the oxide, as an ordinary Bunsen takes a considerable time to oxidise even a hundred or so milligrams of the metal.

To show the degree of accuracy which may be obtained under such circumstances, 101'7 m.grms. of Cu were obtained by placing 105'2 m.grms. of zinc in a warm solution of copper sulphate, which gives an equivalent of 62'8 for the copper.

I have made a large number of experiments with magnesium and copper sulphate and chloride, using both dilute and concentrated solutions, at about 18°, 50°, and 90° C., also solutions made alkaline with ammonia and sodium hydrate, but invariably with unsatisfactory results. The copper or copper oxide obtained varies greatly in amount, but is never much above 70 per cent of the theoretical quantity. The highest results were given by hot concentrated solutions; in dilute or cold solution the process required hours to complete, and the results were under 50 per cent. In every case a considerable amount of hydrogen was generated, and more or less of the magnesium became oxidised. The magnesium oxide in turn displaced copper hydrate, so that a greenish sediment was always found accompanying the precipitated copper. This result by itself would be enough to prove that the reaction was not a quantitative one. Very dilute hydrochloric acid was used to dissolve this greenish substance, and a small quantity of the copper was removed at the same time (not more than 8 to 10 per cent).

* We showed by later experiments, described further on, the influence of the presence of chloride of ammonium; we added it in a large number of experiments, so as to get as near as possible to the conditions of mineral analysis.

I give one or two of my results:—

Mg.	Cu.	Cu (theoretical).	
0.055 grm.	0.079	0.144	Strong solution at about 50° C. One hour required.
0.055 "	0.064	0.144	Strong solution at 18° C. Several hours.
0.065 "	0.137	0.171	Conc. sol. at about 80° C. A few minutes only.

The results obtained from perfectly pure magnesium might be very different from the above, but I have not been able to use any but the commercial metal.

An excess of magnesium oxide removes every trace of copper from solutions of the sulphate and chloride, and a bluish residue is obtained containing copper hydrate (?) and magnesium oxide. It settles readily, filters easily, and is unchanged by long boiling, or by exposure to air or water for some weeks.

ANALYSIS OF BRONZES AND BRASSES BY THE ELECTROLYTIC PROCESS.

By A. HOLLAND.

THE object of this communication is to give in detail the procedures for the accurate and easy determination of copper, tin, zinc, &c., entering into the composition of bronzes and brasses.

I. Bronzes.

Determination of Copper (Electrolysis in Acid Solution).—Five grms. of alloy are treated in a beaker (Bohemian glass) with a mixture of 25 c.c. nitric acid, at sp. gr. 1.31, and 15 c.c. concentrated sulphuric acid. In presence of so large a proportion of sulphuric acid the tin dissolves, at least in part. We dilute to 350°, and heat the liquid to a temperature close upon ebullition, keeping it at this temperature until the insoluble part which contains the tin has collected at the bottom of the vessel. We thus obtain a perfectly clear liquid, into which we can plunge without causing turbidity, the platinum cone and spiral serving as electrodes. For the progress of the electrolysis we follow the indications given in *Comptes Rendus*, cxxiii, p. 1003.

Determination of Tin (Electrolysis in Hydrochloric Solution, with the addition of Ammonium Oxalate).—The liquid, free from copper, is evaporated on the sand-bath until there remain only a few drops of sulphuric acid. The residue is taken up in hydrochloric acid and water, and the tin is precipitated by a current of sulphuretted hydrogen in the ordinary manner. The tin sulphide, washed as usual with a solution of sodium chloride, is dissolved in yellow ammonium hydrosulphate, and this solution is evaporated to dryness on the water-bath. The residue obtained is attacked with 9 grms. of potassium chlorate dissolved in water and an excess of hydrochloric acid. The solution of tin thus obtained is again evaporated to dryness on the water-bath, and the residue taken up in 30 c.c. of hydrochloric acid and water. We filter this new solution, and dissolve in it 30 grms. of pure ammonium oxalate; and lastly, heated to about 90° and electrolysed, using a current of 0.7 ampère. At the end of twelve hours the deposition is complete, and the deposit is strongly adhesive.

Determination of Zinc by Electrolysis.—The liquid, free from copper and tin, is heated to expel all the hydrogen sulphide in solution, then evaporated to dryness on the sand-bath until there remain only a few drops of sulphuric acid. The zinc sulphate thus formed is dissolved in water, neutralised with ammonia, and to the solution there are added 15 grms. of ammonium citrate and 4 c.c.

glacial acetic acid, ammonia up to neutralisation, and, lastly, 3 c.c. crystallisable acetic acid.

The bath thus obtained contains, besides zinc in the state of sulphate, ammonium, acetate and citrate, and acetic acid. It is exposed for about twelve hours to a current of 0.6 ampère. At the end of this time all zinc is deposited upon the cone as a very adhesive deposit.

If the bronze contains iron, this will be deposited with the zinc, in part at least. In this case the weight of the iron (determined by permanganate) is deducted from the weight of the zinc thus found.

Lead, which is often met with in bronzes, is determined by electrolysis in a fresh nitric solution.

II. Brasses.

Copper is determined as in *Comptes Rendus*, cxxiii, p. 1003. The determination of zinc and of impurities is effected by the procedures given above.—*Comptes Rendus*, cxxiv, p. 1451.

ON CERTAIN DOUBLE HALOGEN SALTS OF CÆSIUM AND RUBIDIUM.

By H. L. WELLS and H. W. FOOTE.

1. The Complicated Rubidium-antimony Chloride.

REMSEN and Saunders (*Am. Chem. Journ.*, xiv., 155) have described a salt to which they gave the formula $23\text{RbCl} \cdot 10\text{SbCl}_3$ as the most probable one. Wheeler (*Am. Journ. Sci.*, xlv., 269), working in the Sheffield Chemical Laboratory, New Haven, confirmed Remsen and Saunders's results and discovered besides an analogous bromide, to which the probable formula $23\text{RbBr} \cdot 10\text{SbBr}_3$ was given. Remsen and Brigham (*Am. Chem. Journ.*, xiv., 174) prepared the salt $23\text{RbCl} \cdot 10\text{BiCl}_3$. Hery (*Am. Chem. Journ.*, xvi., 490) has since described the two potassium salts, $23\text{KCl} \cdot 10\text{SbCl}_3$ and $23\text{KBr} \cdot 10\text{SbBr}_3 \cdot 27\text{H}_2\text{O}$, and some mixtures of these two salts.

In view of all this work there can scarcely be a doubt as to the existence of a type of salts with a somewhat complicated ratio, but in view of the fact that this complicated ratio 23:10 is apparently an exception to the simplicity of composition of all other carefully investigated double halogen salts, the subject seemed worthy of some further investigation. For the purpose, we have studied only the rubidium-antimony chloride of Remsen and Saunders, as this salt is readily prepared and is capable of repeated re-crystallisation from hydrochloric acid solution.

The possibility suggested itself that the product might consist of two simpler salts of similar or identical crystalline form, which were capable of crystallising together, and that previous investigators had made use of conditions which resulted in obtaining a constant mixture of two such salts. Although this supposition had scarcely any probability in view of the existence also of the rubidium-antimony bromide and of the two potassium salts, we have put the question to test by repeatedly re-crystallising the salt, using not only ordinary dilute hydrochloric acid for this purpose, but also more dilute and much more concentrated acid and also an alcoholic hydrochloric acid solution. As will be seen from the analyses, given beyond, no variation in composition could be detected by the use of these widely varying solvents for re-crystallisation, and it therefore appears impossible that the salt can be a mixture.

As a starting-point, we used a solution in hydrochloric acid containing the constituents RbCl and SbCl_3 in the exact molecular proportion 23:10. Product A was the first, B the third, and C the fifth re-crystallisation from pure dilute hydrochloric acid. The product D was obtained by adding concentrated hydrochloric acid to a

nearly saturated warm solution of the salt in dilute hydrochloric acid. E was obtained from a very strong hydrochloric acid solution formed by passing a rapid current of hydrogen chloride gas into the solution as it cooled. F was obtained by re-crystallising the salt from hydrochloric acid which was kept as dilute as it could be without producing the basic double salt to be described beyond. G was a product obtained by re-crystallising the salt from a mixture of equal volumes of dilute hydrochloric acid and alcohol.

The two products obtained from concentrated hydrochloric acid solution had a pale yellow colour, while the others were all white. The crystals were usually well-formed six-sided plates which showed no definite optical properties.

The analyses of the various products are as follows:—

	Rubidium.	Antimony.	Chlorine.
A	39'23	23'85	37'01
B	39'23	23'84	36'99
C	—	23'91	—
D	39'25	23'98	—
E	39'31	23'89	—
F	39'03	23'86	—
G	39'11	23'90	—
Average	39'19	23'89	37'00

Method of Analysis.—For the determination of antimony and rubidium, a portion of about $\frac{1}{4}$ grm. was dissolved in water and enough hydrochloric acid to prevent antimony oxychloride from precipitating. The solution was heated to boiling and hydrogen sulphide passed in. The solution was then cooled, and the antimony sulphide filtered on a Gooch crucible and washed with water and with alcohol. The crucible was then slowly heated to 230° and cooled in an oven filled with carbonic acid. The precipitate was weighed as Sb_2S_3 . The filtrate containing rubidium was evaporated with sulphuric acid and the residue ignited in a stream of air containing ammonia, and weighed as Rb_2SO_4 . Chlorine was determined by dissolving a separate portion in water acidified with tartaric and nitric acids and precipitating with silver nitrate. This was allowed to stand for some time and the precipitate was then collected on a Gooch crucible and weighed. The methods used are almost identical with those of Wheeler.

The accuracy of the antimony determination was checked in the following manner:—The salt, $\text{Cs}_3\text{Sb}_2\text{Cl}_9$, was prepared from very pure materials and carefully re-crystallised, and antimony determined by the above method. The per cent of antimony is nearly the same as in the rubidium-antimony salt under consideration. The following results were obtained:—

	I.	II.	III.	IV.
Per cent Sb found	25'37	25'42	25'43	25'44
„ „ calculated	25'13	—	—	—

The atomic weights used in all the calculations were Rb, 85'43; Sb, 120'43; Cl, 35'45; S, 32'07; Ag, 107'92; Cs, 132'89.

Since the method used for the determination of antimony gives results which are slightly too high, we believe that a deduction of the average error 0'25 per cent from the antimony found in the analyses of the rubidium salt will give a result which is nearer the truth.

	Rb.	Sb.	Cl.
Average previously given	39'19	23'89	37'00
Average with correction for Sb	39'19	23'64	37'00
Calculated for $\text{Rb}_{23}\text{Sb}_{10}\text{Cl}_{53}$	38'92	23'86	37'22
Calculated for $\text{Rb}_7\text{Sb}_3\text{Cl}_{16}$	39'18	23'66	37'16

It may be noticed that the results agree rather more satisfactorily with the formula $7\text{RbCl}_3\text{SbCl}_3$ than with the more complicated one advanced by Remsen and Saunders. The differences between these formulæ are,

however, so slight that it is probably entirely impossible to decide between them by means of chemical analysis, the ratio Rb : Sb being 230 : 100 in one case, and in the other 233 : 100. However, since it is customary to use the simplest applicable formula for a chemical compound, we propose the formula $7\text{RbCl}_3\text{SbCl}_3$ for this salt, and corresponding formulæ for other salts of this series. Herty's hydrous salt, to which he gave the formula $23\text{KBr} \cdot 10\text{SbBr}_3 \cdot 27\text{H}_2\text{O}$, agrees well with the formula $7\text{KBr} \cdot 3\text{SbBr}_3 \cdot 8\text{H}_2\text{O}$. It must be admitted that the 7 : 3 ratio is an unusually complicated one, but it is far simpler than 23 : 10, and is scarcely a marked exception to the general simplicity of double halogen salts.

2. A Rubidium-antimony Oxychloride, $2\text{RbCl} \cdot \text{SbCl}_3 \cdot \text{SbOCl}$.

In attempting to re-crystallise the salt $7\text{RbCl}_3\text{SbCl}_3$ from very dilute hydrochloric acid, just enough to prevent the formation of antimony oxychloride, this new salt was obtained in the form of short colourless prisms possessing a rather high lustre. It can be re-crystallised from very dilute hydrochloric acid.

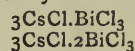
The following results were obtained from analyses of separate crops:—

	I.	II.	Calculated for $2\text{RbCl} \cdot \text{SbCl}_3 \cdot \text{SbOCl}$.
Rb	26'54	26'68	26'68
Sb	37'58	37'36	37'61
Cl	32'75	32'80	33'21
O (by diff.) ..	3'13	3'16	2'50

It is interesting to notice that Benediç (*Proc. Am. Acad.*, xxix., 212) has described the potassium salt $2\text{KCl} \cdot \text{SbCl}_3 \cdot \text{SbOCl}$, which corresponds exactly to this rubidium compound.

3. The Cæsium-bismuth Chlorides and Iodides.

The double chlorides of bismuth with cæsium have been described by Remsen and Brigham (*Am. Chem. Journ.*, xiv., 179). These authors did not state, however, how widely the conditions had been varied, and we have repeated the work, varying the proportions of cæsium and bismuth as much as possible, and have found exactly the same salts as described by them. These salts are,—



$3\text{CsCl} \cdot \text{BiCl}_3$.—This salt forms in colourless plates when 50 grms. of cæsium chloride are mixed in hydrochloric acid solution with from 1 to 25 grms. of bismuth chloride. The analyses were made on samples, dried but a short time in the air, which apparently contained a little mechanically included water. The following results were obtained:—

	I.	II.	Calculated for $3\text{CsCl} \cdot \text{BiCl}_3$.
Bi	24'80	24'47	25'36
Cs	47'94	—	48'66
Cl	—	—	25'98

$3\text{CsCl} \cdot 2\text{BiCl}_3$.—When 50 grms. of bismuth chloride are mixed with from 1 to 80 grms. of cæsium chloride, the salt $3\text{CsCl} \cdot 2\text{BiCl}_3$ crystallises in light yellow needles, sometimes broadening and looking like plates and again much shorter and thicker.

The following analyses were made:—

	I.	II.	Calculated for $3\text{CsCl} \cdot 2\text{BiCl}_3$.
Bi	36'99	36'58	36'67
Cs	34'69	34'94	35'17
Cl	—	—	28'16

$3\text{CsI} \cdot 2\text{BiI}_3$. Cæsium-bismuth Iodide.

We could obtain only one double iodide of bismuth and cæsium, although the proportions of cæsium and bismuth were varied greatly. The salt formed as a crystalline precipitate, difficultly soluble especially in an excess of

cæsium iodide, when 1 grm. of bismuth iodide was added to 50 grms. of cæsium iodide, and when 1 grm. of cæsium iodide was added to 50 grms. of bismuth iodide. With an excess of cæsium, the colour was a bright red, while with an excess of bismuth the colour was more of a reddish brown.

Methods of Analysis.—The methods here given were used in both the double chlorides and iodide of bismuth.

Halogens were determined as the silver salts, being precipitated from a solution acidified with tartaric and nitric acids, and, after standing, filtered and weighed on a Gooch crucible. As Remsen and Brigham had mentioned a difficulty in determining bismuth, we made a few determinations of it in Bi_2O_3 , which was made by precipitating BiONO_3 with water from a nitric acid solution of $\text{Bi}(\text{NO}_3)_3$, and heating the precipitate to constant weight in a platinum dish. The method finally adopted was to dissolve the substance in water slightly acidified with hydrochloric acid and precipitate Bi_2S_3 from the cold solution with hydrogen sulphide. The precipitate was filtered and immediately dissolved in nitric acid and digested for some time on the water-bath until completely decomposed. The sulphur was filtered off and the filtrate, diluted to about 300–400 c.c., was heated and ammonium carbonate added in slight excess. It was placed on the water-bath for an hour or two, until the liquid had become nearly clear and the excess of ammonium carbonate had been driven off, and it was then filtered on a Gooch crucible and ignited strongly over a Bunsen burner and weighed as Bi_2O_3 .

Two determinations on Bi_2O_3 gave the following results:—

	Grm.		Grm.
I. Amt. Bi_2O_3 taken =	0.1979	Amt. Bi_2O_3 found =	0.1974
II. " " =	0.3604	" " =	0.3617

The filtrate from the bismuth precipitation was evaporated with sulphuric acid and ignited in a stream of air containing ammonia. The residue was weighed as Cs_2SO_4 .

The results obtained from the analysis of the double iodide were as follows:—

	I.	II.	Calculated for $\text{Cs}_3\text{Bi}_2\text{I}_9$.
Bi	21.34	21.15	21.25
Cs	20.75	20.31	20.38
I	—	58.02	58.37

—*American Journal of Science*, iii., No. 18, June, 1897.

PROCEEDINGS OF SOCIETIES.

CHEMICAL AND METALLURGICAL SOCIETY, JOHANNESBURG.

May 15, 1897.

AFTER a few remarks on Prof. Christy's paper, read at a previous meeting, the Society proceeded to discuss and comment on Mr. EHRMANN's paper on the "*Precipitation of Gold from Cyanide Solutions.*"

The drift of the discussion was as to the advantage, if any, compared with the increased cost, to be gained by heating the solution to promote rapid precipitation; it does not appear to be at all certain whether there is any appreciable quickening of the process during the summer months as compared with the winter, and, as is well known to those who have lived in the country, the range of temperature is very great. The cost of heating large quantities of solution would be very prohibitive.

A few remarks were made on Dr. STOCKHAUSEN's paper on "*The Liqation in Cyanide Bars,*" and Mr. D. J. WILLIAMS then read a few notes on "*The Estimation of Lead in Slags and other By-products.*"

SOCIÉTÉ D'ENCOURAGEMENT POUR L'INDUSTRIE NATIONALE.

June 25, 1897.

M. MASCART, President, in the chair.

AFTER reading the correspondence and hearing the reports of the committees on the Mechanical Arts and the Economic Arts a communication was made by M. CHEYSSON on the "*Insurance in French Industries against Accidents.*" The author was warmly thanked for his paper, which was ordered to be inserted in the *Bulletin*.

The President delivered a long discourse on the progress of the Society, and made special reference to absent and foreign members. A list of medals and prizes awarded was then read, and the meeting adjourned.

NOTICES OF BOOKS.

The Chlorination Process. By E. P. WILSON, E.M. First Edition, first thousand. New York: John Wiley and Sons. London: Chapman and Hall, Limited. 1897. Pp. v—125, 12mo., Ill.

PLATTNER laid the foundation for the process of extracting gold from its ores by the agency of chlorine, in 1856, and since then the method has undergone many improvements. The first to make a practical and commercial success of chlorination was Mr. G. F. Deitken, in California. Mears and Theis subsequently replaced tank-lixiviation by barrel-chlorination, and the latter method of treatment prevails.

Mr. Wilson has written very clearly and explicitly, showing familiarity with the literature of the subject, and with the practical workings of many American works. In successive chapters he describes the preparation of the ore, the methods of roasting, the furnaces, the leaching process, filtering methods, precipitation, and the refining of the precipitated gold; the final chapter is devoted to the cost of chlorination.

Mr. Wilson shows a great deal of common sense in his occasional remarks, as in the following paragraph:—"Some wise person has stated that 'all is not gold that glitters,' and, if he were alive and a miner, he could have added two other facts which history has established, viz., that all gold-bearing rocks do not contain gold in paying quantities; also, that some gold-bearing rocks contain considerable quantities of gold, but are commercially valueless."

Due consideration of these statements might have prevented great losses in gold-mining ventures.

Again he writes:—"The chlorination process has not been more generally adopted because those who run mines are not capable of carrying it on, and, knowing their weakness, let it alone. And parties having mining machinery to sell discourage the chlorination process. Lastly, mine-owners, thinking they will have to pay higher salaries to good men, are willing to suffer loss of gold rather than do so."

The book has been written in a style intelligible to mine-owners as well as to students of metallurgy, though the interests of the latter have been regarded by the introduction of chemical equations.

Mr. Wilson, in common with so many writers of English, uses the French word *résumé* where "summary" will serve equally well.

This little work forms a companion volume to the author's "*Cyanide Processes.*" The book is well printed on good paper, and contains an Index.

H. C. B.

The Agricultural Journal. Department of Agriculture, Cape of Good Hope. Vol. x., No. 10, May 13, 1897.

A GREAT deal of this number is taken up with matter purely of interest to farmers and stock-breeders; some attention is being paid to artificial manuring, as the farmers there are beginning to realise that, unless the production can be increased, farming will not pay, and that what is taken from the soil must be returned to it.

There are some long extracts from Dr. Wm. Newton's paper on Nitrates, read before the Liverpool Meeting of the British Association last year; and some further remarks by Dr. Dyer on the use of sulphate of ammonia for the same purpose.

In connection with the experiments being now conducted in the colony for the extirpation of *Tcerya Purchasi* (Australian bug) and scale insects, by the introduction of ladybirds from California and Australia, an instructive communication is produced, showing how entirely successful the same experiment has been in the Hawaiian Islands. The first importation was made in 1890, when *Vedalia cardinalis* was sent over, at the time when most trees were in a deplorable condition from the attacks of *Tcerya*; the *Vedalia* was a complete success. It became perfectly naturalised, increased prodigiously for a time, practically cleared the trees, and then—as the *Tcerya* became comparatively scarce—decreased in numbers, while at the present time it is evident that the number of the scale insect and its destroyer has arrived at a fixed proportion. The ladybird has previously done excellent service in the fruit orchards in Lower California. The effect is not imaginary, but proven. In June, 1895, a lovely forest in Hawaii—5000 feet above sea-level—was found to be much affected by a black *Aphis*. By beating the trees the blight came down in abundance. One or two introduced ladybirds were also noticed. By September the ladybirds were present in thousands,—the blight and native insects in small numbers. In August, 1896, not an *Aphis* was to be found, and only one or two stray ladybirds. They had done their work and disappeared.

Thirty-third Annual Report on Alkali, &c., Works. By THE CHIEF INSPECTOR. London: Eyre and Spottiswoode. 1897.

FOLLOWING the appointment of an additional Sub-inspector, there has been a re-distribution of district areas, which has relieved the work in some of the districts. The number of Works now registered in England, Ireland, and Wales under the A&T is 1074. Of these 98 only are works decomposing salt, and so scheduled as Alkali Works, while the remainder, 976, carry on processes which are scheduled under the A&Ts of 1881 and 1892. These numbers show a decrease of three alkali works and an increase of twelve other works since 1895. There are also 125 works registered in Scotland, bringing the total number registered to 1199.

There has been one prosecution for "obstruction" under Section 17 of the A&T of 1881. The important point was that, after the works had been passed for registration, on the basis of plans supplied by the proprietor, the manager had carried out the deception of the by-pass pipes, which prevented the Inspector from examining the effluent gases. The works in question, for the manufacture of carbon bisulphide, are now closed, and it is not likely the manufacture will be resumed. In addition, there were five cases of unregistered works being carried on; but in each case the fees were accepted by the Board, it appearing that ignorance existed of the obligations of the A&T.

The amount of salt decomposed in the Leblanc process again shows a reduction, and one more considerable than has occurred since 1893. On the other hand, the ammonia-soda process has largely increased its lead over its rival, obtained for the first time last year.

A new departure in the manufacture of chlorate of soda, a salt much used by calico-printers, has taken place during the year, by the introduction, on the large scale, of Mr. J. Hargreaves's process for chlorinating hydrated sodium carbonate directly, in an absorbing tower, in which lixiviation of the products to remove the sodium chloride is also conducted.

There has been an increase in the amount of ammonia recovered and made, but it is anticipated that in a few years there will be a decrease, owing to the extensive adoption by gas engineers of carburetted water-gas plant in connection with gas manufacture in many parts of the country. In America coal-gas has been displaced to the extent of over 70 per cent by carburetted water-gas; in this manufacture no ammonia is produced.

General Index to the Proceedings of the Society of Public Analysts. Compiled by J. C. WELCH, F.C.S. London: Baillière, Tindall, and Cox. 1897.

THE compiler originally commenced this work for his own private convenience, but the allusion to this subject made by Mr. Otto Hehner, in his Presidential Address in 1893, led him to offer the manuscript to the Society when completed to the end of the twentieth volume.

The thanks of public analysts generally are due to Mr. Welch for the care he has taken and the time he has expended in the laborious task.

CORRESPONDENCE.

THE PREPARATION OF ZINC ETHYL.

To the Editor of the Chemical News.

SIR,—In the last issue of the CHEMICAL NEWS (vol. lxxvi., p. 20) there appears under this heading an abstract of a paper by A. Lachman from the *American Chemical Journal*. Mr. Lachman employs a copper-zinc couple, which he prepares by mixing zinc-dust with copper oxide, and then reducing the copper by passing hydrogen over the heated mixture. For many years I have been in the habit of preparing zinc ethyl, using a copper-zinc couple consisting of a mixture of zinc dust and copper oxide, *without reducing the copper oxide to the metallic state*, and a description of the process was published in my "Chemical Lecture Experiments" in 1892. Such a mixture acts upon ethyl iodide with extreme readiness; I have performed the entire operation of preparing zinc ethyl and distilling it off during a lecture of an hour.—I am, &c.,

G. S. NEWTH.

Royal College of Science, London.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 24, June 14, 1897.

Contribution to the History of the Phosphorus Iodides.—A. Besson.—The solution appears to contain an instable compound, P_3I_4 , which is the pivot of the apparent transformation of white phosphorus into red phosphorus by the action of iodine.

Procedure of Oxidation and Chloridation.—A. Villiers.—When an oxidisable body occurs in a medium capable of furnishing oxygen, but under such conditions

that the oxidation either does not begin at all or takes place very slowly, the addition of a trace of a salt of manganese determines the reaction or accelerates it considerably.

Splitting up of the Fundamental Band of the Chlorophylls.—A. Etard.—The number of chlorophyll bands and the wave-length of their mean axis may, by the method of limited dilutions, be counted exactly, and may serve to characterise the chemical species. The diversity of the chlorophylls is demonstrated by the wave-length of the axes of their bands, whether pre-existing or induced by the action of reagents. The fundamental band of the chlorophylls is not always uniformly obscure; it may be double or triple.

On the Oxidising Action of Manganese Salts, and on the Chemical Constitution of the Oxidases.—G. Bertrand.—All the manganous salts which the author has tried possess the property of fixing free oxygen upon hydroquinone. They behave in the same manner with pyrogallol, paramidophenol, and other kindred bodies.

Action of Nickel upon Ethylene. Synthesis of Ethane.—Paul Sabatier and J. B. Senderens.—If we direct a mixture of equal vols. of ethylene and hydrogen upon nickel recently reduced and slightly heated (30° to 45°), we observe a notable rise of temperature, owing to the formation of ethane; copper, iron, and cobalt cannot serve to effect this synthesis.

Isolaunonic Acid.—G. Blanc.—This memoir will be inserted at length if possible.

Action of Acetylene upon Silver Nitrate.—R. Chavastelon.— C_2H_2 forms with silver nitrate, according to the nature of the solvent, $C_2Ag_2.NO_2Ag - C_2Ag_2$. In future communications the author will describe a procedure for the determination of acetylene applicable in a great number of cases. He will also study the crystalline compounds of acetylene with cuprous chloride and mercuric chloride.

Determination of Resin Oil in Oil of Turpentine.—A. Aignon.—The author gives his results in the form of a table. It is possible, even on rectifying at 100° under a pressure reduced to 0.06 metre, to obtain a dextro-rotatory residue. The pure oil, if treated in the same manner, gives only lævo-rotatory residues.

Active Principles of some Aroids.—Mlle. Chauliaguet, A. Hébert, and F. Hein.—The authors have not been able to detect the presence of hydrocyanic acid. The symptoms of poisoning by arums and the appearances observed on autopsy do not resemble those occasioned by hydrocyanic acid.

Action of Albumoses and Peptones on Inter-vascular Injection.—E. Fiquet.—The author's results are interesting physiologically rather than chemically.

Revue Universelle des Mines et de la Metallurgie.
Series 3, Vol. xxxviii., No. 2.

This issue contains no matter of chemical interest.

Revue Générale des Sciences Pures et Appliquées.
No. 10, May 30, 1897.

This number contains no original matter of special chemical interest.

Journal de Pharmacie et Chimie.
Series 6, vol v., No. 11.

Contribution to the Study of the Preparation of Ordinary Ether.—L. Prunier.—In the study of the preparation of ordinary ether by means of sulphuric acid and alcohol, most workers have omitted to take notice of the presence of sulphonic acids and their derivatives. This group of bodies is, however, to be found in notable

quantities in commercial ethers. It is also found in considerable proportions in the oils which have been used in the rectification of the raw product. By direct experiment it is possible to prove the formation of several sulphonic derivatives, especially towards the end of the operation. To separate the derivatives actually formed by the action of sulphuric acid, it suffices to heat sulphovinic acid with dilute sulphuric acid to 140° , then add a little alcohol. By this means a small quantity of ordinary ether is formed, and several sulphonic derivatives of varying volatilities, some of which even distil over with the ether. They are formed in greatest abundance when the temperature exceeds 140° , and above all if undiluted sulphuric acid be used. It is exactly the same as in the preparation of ethylene; if the operation be interrupted when the liquid becomes dark (165° to 175°) we can at that moment detect the presence of a large quantity of sulphonic compounds which exist in the liquid, along with the sulphuric acid in excess, traces of sulphovinic acid neutral sulphuric ether, polyethylenic compounds, and also sulphurous acid.

Researches on the Composition of Extracts of Meat.—J. Bruylants.—It has long been admitted that extracts of meat contain, as proteic substances, but a small quantity of gelatin. A large proportion of the nitrogen belongs to other proteic substances, albumoses, and peptones; that is to say, to substances whose nutritive value is greater than that of albumides, since they have already undergone some of the modifications due to digestion. The result of the examination of a number of different samples of extracts shows that Liebig's extract is the richest in the most nutrient and assimilable substances, though dry Bovril is not far below it; liquid Bovril is comparatively poor.

Method of Estimating Aldehyd in Ether.—L. Francois.—Already inserted.

Different varieties of Chestnuts.—M. Balland.—Not suitable for abstraction.

Reaction enabling Naphthol- α to be easily Distinguished from Naphthol- β .—E. Léger.—On April 7th the author made a communication on the action of hypobromite of sodium on certain phenols. In view of the facts therein described, he prepares from the supposed mixture a saturated aqueous solution; this solution, when diluted with its own volume of water, will not give the naphthol- β reaction with 2 drops of hypobromite, but if the substance under examination contain naphthol- α we get a violet or violet-rose colouration. By this means it is easy to detect 1 part of naphthol- α in 100 parts of naphthol- β .

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xvii.-xviii., No. 11. June 5, 1897.

Action of Hydrate of Chloral on Phenylhydrazine. Diphenylglyoxazol and its Derivatives.—H. M. Causse.—Hydrate of chloral unites very easily with phenylhydrazine, forming as a rule a crystalline compound, but sometimes an oil denser than water is produced; the crystals, however, are very unstable, so much so that analysis is impossible, the spontaneous decomposition being so rapid. The author has therefore been obliged to pass over the intermediary compounds and confine his research to the final products.

Monobromated Camphor.—Ch. Moureu.—Monobromated camphor is possessed of a remarkable stability; the brown appears to be united to the carbon as firmly as in aromatic bodies. Heated to 200° with phosphoric anhydride, an energetic reaction takes place, tarry products are formed, an abundance of gas is given off, and a liquid—fuming very strongly in contact with air—is distilled over. This latter has been found to be tribromide of phosphorus, the formation of which is of great interest.

MISCELLANEOUS.

Iron and Steel Institute.—The autumn meeting of the Iron and Steel Institute will be held at Cardiff, under the presidency of Edward Martin, Esq., on the 3rd, 4th, 5th, and 6th of August next. The following papers will be read, and members wishing to take part in the discussions will, on application to the Secretary, Mr. B. H. Brough, have copies of the papers forwarded to them a week in advance, as far as that may be possible:—

- "On Passive Iron." By J. S. de Benneville (Philadelphia).
- "On the Diffusion of Sulphides through Steel." By E. D. Campbell (Ann Arbor, Michigan).
- "On the Manufacture of Tin Plates." By George B. Hammond (Penarth).
- "On a Spectroscopic Analysis of Iron Ores." By Prof. W. N. Hartley, F.R.S., and Hugh Ramage, Assoc. R.C.Sc.I., F.I.C. (Royal College of Science, Dublin).
- "On Improvements in Shipping Appliances in the Bristol Channel." By Sir W. T. Lewis, Bart. Member of Council.
- "On the Iron Industry of Hungary." By D. A. Louis, F.I.C. (London).
- "On a Thermo-chemical Study of the Refining of Iron." By Prof. Honoré Ponthière (Louvain).
- "On Carbon and Iron." By E. H. Saniter (Wigan).
- "On some Mechanical Appliances at Penarth Docks." By T. Hurry Riches, M.Inst.C.E. (Cardiff).
- "On the Application of Travelling Belts to the Shipment of Coal." By Thomas Wrightson, M.Inst.C.E. (Thornaby-on-Tees).

There will be, as usual, a number of excursions to works and other places of interest in the neighbourhood, particulars of which, together with the full programme of the meeting, may be obtained from the Secretary, at 28, Victoria Street, London, S.W.

Combustion of Nitrogen.—O. Bleier (*Berichte*).—Nitrogen is mixed with oxygen in the proper proportion, introduced into an enamelled autoclave, or other suitable vessel, containing dilute alkali, and an excess of detonating gas is pumped in. The mixture is exploded, and the oxides of nitrogen are removed by shaking up with alkali. Detonating gas is again introduced, and the process is repeated.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 5th inst., Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, presiding. The following were elected Members:—H. H. Baird, Ivon Braby, J. M. Davidson, M.B., C.M., A. C. Hill, B.A., J. Y. Johnson, L. Kamm, M. E. Stephens, The Rev. Henry Wace, D.D., Julius Wernher, and Henry Wilde, F.R.S. The Special Thanks of the Members were returned to Sir Andrew Noble for his donation of £100 to the Fund for the Promotion of Experimental Research at Low Temperatures.

NOTES AND QUERIES.

*** Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Marking Inks.—(Reply to "Sulpho").—Ample information upon the subject, with the latest recipes, will be found in the Second Series of Spon's "Workshop Receipts," 1890.—GEORGE TURNER, 5, South Street, E.C.

Mr. J. G. LORRAIN, M.I.E.E., M.I.M.E., M.S.C.I.,
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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1965.

MIGRANT MATTER.

By STEPHEN H. EMMENS and NEWTON W. EMMENS.

On the 5th of March, in this year, we cut a circular disc, 1 inch in diameter, from a thin sheet of lead purchased as being "chemically pure" and showing no residual metal on cupellation. We also cut a similar disc from a thin sheet of silver purchased as being "chemically pure." These discs were then weighed and placed in contact with the two ends of a short spiral of copper wire of known weight; and the system thus composed was suspended inside a wide-mouthed glass bottle by means of a string depending from the cork and passing through the spiral and central holes in the discs. The silver disc formed the lowest member of the system.

On the 15th of March the bottle, which had meanwhile stood on one of the shelves in our laboratory, was opened, and the discs and spiral were taken apart and weighed. They were then replaced as at first, and allowed to remain untouched until the 2nd inst., when they were again weighed.

The figures (in grms.) of the three weighings were as follows:—

	Lead.	Silver.	Copper.
March 5 ..	0·8931	0·50845	1·5421
" 15 ..	0·8932	0·50830	1·5420
June 2 ..	0·8932	0·50810	1·5421

We dissolved the copper in nitric acid. The solution on being tested with hydrochloric acid, gave no reaction for silver. But the lead disc, on cupellation, gave a silver bead weighing 0·00003 grm.

It would appear, from this experiment, that what is commonly recognised as solid silver is, in part at least, a migrant mode of matter. It would also appear possible that the gold found by Prof. Roberts-Austen in cylinders of lead which had been standing for some time upon golden bases may have migrated from the outer surface of such bases instead of travelling through the interior of the metallic column. The atmosphere certainly seems to be the path of least resistance.

We use the term "migrant matter" because the travelling particles to which we refer are (in common with odours generally) much more akin to Crookes's "fourth form" than to gases.

Argentaurum Laboratory,
June 5, 1897.

ON THE ESTIMATION OF OXYGEN DISSOLVED IN SEA-WATER.

By ALBERT LÉVY and F. MARBOUTIN.

SOME time ago one of us published the details of a precise and rapid method of estimating the oxygen dissolved in waters. It having been in constant use for fifteen years at the Observatory at Montsouris, we have been able to follow, week by week, the variations existing, from the point of view of oxygen dissolved, in waters from springs, rivers, and drainage. The method consists in partially peroxidising, by the help of the oxygen dissolved in the water, an excess of protoxide of iron, and completing the oxidation by means of a titrated solution of permanganate

of potash. We have described in the different *Annals* of Montsouris the method of procedure, and have each year discussed the results obtained. This method, however, presented several difficulties when we wished to apply it to the analysis of waters containing much chlorides, such as sea-water. In this case, at the moment of adding the permanganate, we observe a disengagement of chlorine which conduces to a too high reading:—

50 c.c. fresh water, reading 22·80 c. c. No chlorine.
50 c.c. fresh water + 50 c.c. of chloride solution, reading 23·00 c.c. Cl given off.
50 c.c. fresh water + 100 c.c. of chloride solution, reading 23·30 c.c. Cl given off.

We can, however, even with waters high in chlorides, succeed in getting good results with permanganate, by making a special datum of comparison for each water and agitating the liquid cautiously, and always working in an identical manner.

But we prefer, for such waters high in chlorides, to replace the permanganate by bichromate of potash, and to determine the end of the operation, by means of the touch test, with ferrocyanide of potassium. We have made the following observations:—

1. In the case of a spring or river water, the permanganate and the bichromate give precisely the same result, and this result is identical with that obtained by extracting the gas with a mercury pump.

Eau d'Aure, taken 10th March, 1897, from the Reservoir in the Rue Villejust.

Permanganate method: volume of water 95·2 c.c.
Datum .. 24·00 c.c. Reading .. 17·30 c.c.

In 1 litre of water,—
 $\frac{24·00 - 17·30}{95·2} \times 160·8$ 11·32 m.grms.

Bichromate method: volume of water 96·6 c.c.
Datum .. 23·80 c.c. Reading .. 17·00 c.c.
In 1 litre of water,—
 $\frac{23·80 - 17·00}{96·6} \times 160·0$ 11·26 m.grms.

Oxygen extracted by the mercury pump:—Amount operated on, 364·8 c.c. Oxygen measured at 0° C. and 760 m.m., 2·872 c.c.

In 1 litre of water,—
 $1·43 \times \frac{2·873 \times 1000}{364·8}$ 11·26 m.grms.

2. The oxygen dissolved in sea-water is estimated very exactly by the use of bichromate, in spite of the large quantity of chlorides and magnesian salts.

We worked on a sample taken in a carboy, sent from Concarneau by the kindness of M. Fabre-Domergue.

Three determinations by means of bichromate gave, per litre of water,—

1st. $\frac{23·50 - 17·40}{102·6} \times 160·0$.. 9·52 m.grms.

2nd. $\frac{23·50 - 17·65}{98·3} \times 160·0$.. 9·52 m.grms.

3rd. $\frac{23·50 - 17·75}{96·6} \times 160·0$.. 9·53 m.grms.

Oxygen extracted with the mercury pump: Amount operated on, 364·800 c.c. Oxygen measured at 0° C. and 760 m.m., 2·432 c.c.

In 1 litre of water,—
 $1·43 \times \frac{2·432 \times 1000}{364·8}$ 9·51 m.grms.

When waters such as sea-water contain much magnesia, at the moment, following our method, when we make the liquid alkaline with potash, we see the magnesia precipitated in the form of little cylinders similar to grains

of rice. This precipitate in no way interferes with the success of the operation; it is only necessary to turn the tube holding the liquid up and down a few times, so that the precipitate may be equally disseminated throughout the solution.

Bichromate retains its titration almost indefinitely.—*Bull. Soc. Chim.*, Series 3, vol. xvii.-xviii., No. 12.

ON THE VOLUMETRIC DETERMINATION OF ZINC BY POTASSIUM FERROCYANIDE.

By L. L. DE KONINCK and EUG. PROST.

(Continued from p. 30).

THE two series were done at an interval of several days, and with different ferrocyanide solutions; that used for the first series (Solution A) was prepared from salt purified by crystallisation, the other (Solution B) from ordinary commercial ferrocyanide. It may be noted that the figures of the second series diminish more regularly than those of the first; that is caused by the reason, that in the second series the titration back was not done until after digestion for from twenty to forty-five minutes; while in the first, no account was kept of the time—its influence on the result being not then known to us.

One sees that the influence of chloride of ammonium is very marked; the result differs considerably according to whether the solution does (Nos. 3 to 8) or does not (Nos. 1 and 2) contain any, but within the ordinary limits of practical working (Nos. 3 and 4) there does not seem to be much influence. It will evidently be necessary in making estimations in the presence of chloride of ammonium to work in such a manner that the quantity of this salt should be as near as possible constant, and to titrate the ferrocyanide in presence of a similar quantity.

We are unable to find any explanation of the influence of chloride of ammonium in showing a higher relation between the ferrocyanide and the zinc. At first sight one might think, as with hydrochloric acid, that ferrocyanide being slightly soluble in the reagent in question—in this case a salt of ammonia—an excess of ferrocyanide would be necessary to produce a complete precipitation; but this is not so, as a very simple experiment shows.

By dropping 2 drops of $\frac{1}{2}$ normal ferrocyanide into 15 c.c. of water to which has been added 1 drop of a $\frac{1}{4}$ th normal solution of neutral zinc salt, a hardly noticeable cloudiness is produced, while if we repeat the experiment, but substitute a 20 per cent solution of ammonium chloride for the water, we get a very distinct cloudiness. On the other hand, if we divide the liquid of the first experiment into two equal parts, then add to one 10 c.c. of water, and to the other 10 c.c. of 20 per cent ammonium chloride, we notice that the latter becomes distinctly cloudy, while the former shows no change. Thus, chloride of ammonium favours precipitation.

The end of the reaction is very distinctly marked, especially in Experiments 3 to 6.

Influence of Nitrate of Ammonium.—In the analysis of minerals one is liable to have a small quantity of nitrate of ammonia present, from the fact of using nitric acid in dissolving the sample or for the oxidation of ferrous salts. We thought it advisable to ascertain the influence of this salt.

The experiments were carried out by using 20 c.c. of $\frac{1}{2}$ normal $ZnCl_2$, 50 c.c. of $\frac{1}{4}$ normal A solution of ferrocyanide, 10 c.c. of 5 normal HCl, 50 c.c. of 20 per cent AmCl, and 100 c.c. of water, and titrating back with chloride of zinc after digestion for fifteen minutes.

	AmNO ₃ .	ZnCl ₂ back.	ZnCl ₂ total.
1	0 grm.	4'55	24'55
2	2 grms.	4'55	24'55
3	5 "	4'55	24'55

The end of the experiment was perfectly clearly marked. We see thus that nitrate of ammonia is without any influence whatever within these limits, and we did not think it worth while pushing the experiments any further.

Influence of Hydrochloric Acid.—In these experiments we used 20 c.c. of $\frac{1}{2}$ normal $ZnCl_2$, 50 c.c. of $\frac{1}{4}$ normal ferrocyanide, 50 c.c. of 20 per cent AmCl, and 110 c.c. of water, and 5 normal hydrochloric acid in the proportions shown below:—

	5 normal HCl.	Water.	ZnCl ₂ back.		ZnCl ₂ total.	
			I.	II.	I.	II.
1. ..	10	100	5'00	4'85	25'00	24'85
2. ..	20	90	4'80	4'71	24'80	24'71
3. ..	30	80	4'75	4'56	24'75	24'56
4. ..	—	—	4'60	—	24'60	—
5. ..	40	70	4'40	4'46	24'40	24'46
6. ..	50	60	4'20	4'21	24'20	24'21
7. ..	—	—	4'40	—	24'40	—
8. ..	60	50	4'05	3'91	24'05	23'91
9. ..	70	40	3'75	3'72	23'75	23'72

As in the case of the experiments with chloride of ammonium, we again notice the difference of regularity between the first and second series; the reason of this difference is the same as before; the back titration of the second series was not done until after digesting for twenty minutes in the case of No. 1, to forty minutes for No. 9; while in the first series the back titration was done immediately, but without taking any note of the time elapsing between the addition of the ferrocyanide and the titrating back.

It is obvious that the influence of hydrochloric acid is similar to that of chloride of ammonium; the greater the acidity, the more ferrocyanide there is required to completely precipitate the zinc, or at any rate to show with the indicator.

As we have already remarked with regard to ammonium chloride, it is necessary, in order to obtain very exact results in the estimation of zinc, to use a solution of as near as possible constant acidity for the estimations themselves, as well as in the titration of the ferrocyanide.

Influence of Sulphurous Acid.—A freshly-prepared solution of ferrocyanide of potassium has hardly any colour, but after some time it assumes a yellowish tint. When we make an estimation of zinc with such a solution, we note—after the precipitate has entirely settled—that the liquid also has a distinct yellow colour. This colouration, which Galletti attributed to the solution of zinc ferrocyanide in the acid liquor, is due to ferricyanide. This is easily shown by means of ferric chloride. When, in the analysis of a mineral, it is necessary to treat the solution with hydrosulphuric acid, so as to precipitate the copper, cadmium, &c., we ought to re-oxidise the salts of iron with nitric acid, or bromine, and then only precipitate the iron with ammonia.

When, in our first experiments on minerals, of which we shall speak later on, we used nitric acid for re-oxidation, in the ordinary way, we found that the resulting solution had, after the precipitate had settled, a yellow colouration, considerably more intense than in the case of minerals which had not been subjected to re-oxidation. We were easily enabled to prove that this phenomenon was due to the oxidation of a part of the ferrocyanide by the nitrous acid (Schaffer's reaction, *Sill. Amer. Journ.*, Series 2, vol. xii., p. 117, 1854; see also Van Deventer, *Ber.*, xxvi., p. 589, 1893; and Van Deventer and Jorgens *ibid.*, 932). This latter results from the nitrite of ammonium formed by the action of ammonia on the nitrous compounds produced by the oxidation of the ferrous salts by nitric acid. The ammoniacal filtrate separated from the ferric hydrate precipitate contains a good amount of nitrite, for if we add a little chloride of potassium and then acidulate the solution it shows a distinct colour from the iodine set free.

Similar phenomena to those we have just described are

noticed if we use an excess of bromine to oxidise the ferrous salts, and then precipitate the manganese with the iron by means of ammonia. By the action of the ammonia on the excess of bromine, there is, without doubt, a little hypobromite or bromate of ammonia produced; for on making the liquid acid, we can detect the presence of a small quantity of free bromine, and if we titrate with ferrocyanide under these conditions we note also the formation of ferricyanide. We can, however, get over these difficulties by adding a small quantity of sulphite of soda to the ammoniacal solution before making it acid. Solutions treated in this manner are, after titration and when the precipitate has settled, absolutely colourless.

Before recommending the use of sulphite of soda, we thought it necessary to assure ourselves that it was really innocuous. That we did in the following manner:—We used 20 c.c. of $\frac{1}{2}$ normal ZnCl_2 , 100 c.c. of water, and 15 c.c. of 5 normal HCl. We then added 50 c.c. of $\frac{1}{2}$ normal ferrocyanide (A), and after digesting for fifteen or twenty minutes, we titrated back with zincic chloride.

A. Without the addition of sulphite of soda:—

1. ZnCl_2 back	4.65 c.c.	ZnCl_2 total	24.65
2. " "	4.62 " "	" "	24.62

B. With the addition of 10 c.c. of normal sulphite of soda (12.5 grms. $\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$ per litre) to the zincic solution:—

3. ZnCl_2 back	4.69 c.c.	ZnCl_2 total	24.69
4. " "	4.70 " "	" "	24.70

The solutions, after the sulphide was added, smelt strongly of sulphurous anhydride. The end of the reaction showed more slowly, but as distinctly, with sulphurised solutions as with others, only the brown colouration of ferrocyanide of uranium is less distinct, and, in the case of weak solutions, disappears after a few minutes. It must be remembered that the quantity of sulphite here used is considerable, and corresponds molecularly to the 20 c.c. of $\frac{1}{2}$ normal ZnCl_2 on which the experiments were made, while in ordinary estimations 0.1 to 0.2 c.c. of normal sulphite would be amply sufficient. It is evident that in this proportion its action on the indicators would be nil, as we have conclusively shown.

Influence of Bromine.—Although it would appear evident *a priori* that the adverse influence of the excess of bromine used to oxidise the ferrous salts might be neutralised by means of sulphurous acid, we thought it well to verify the fact by experiment.

1. 20 c.c. of $\frac{1}{2}$ normal ZnCl_2 were made alkaline with 10 c.c. of commercial ammonia, then acidulated with 30 c.c. of 5 normal HCl. We then added 100 c.c. of water, and ran in 50 c.c. of $\frac{1}{2}$ normal ferrocyanide.

2. To 20 c.c. of $\frac{1}{2}$ normal ZnCl_2 we added 15 c.c. of water saturated with bromine, then treated it with ammonia, and acidulated with 30 c.c. of acid; the solution turned a deep yellow. The bulk of the bromine was driven off by boiling, the last traces were got rid of by a few drops of normal sulphite of soda, the liquid was cooled, and 50 c.c. of $\frac{1}{2}$ normal ferrocyanide were added. The back titration with chloride of zinc gave—

1. ZnCl_2 back	4.15 c.c.	ZnCl_2 total	24.45
2. " "	4.50 " "	" "	24.50

The use of bromine has therefore no influence, or rather it is neutralised by the use of sulphite of soda.

Influence of Nitric Acid.—The following experiments were carried out with the object of satisfying ourselves that the addition of sulphite of soda to the zincic solution would neutralise the action of the nitrous acid, which is formed, under the conditions we have already stated, by the oxidation of the ferrous salts with nitric acid. A cuprous and ferruginous calamine, of which five assays were made, either with sodic sulphite or with ferrocyanide and sodic sulphite, showed a proportion of 23.94 to 24.39 per cent of zinc. After treatment with hydrosulphuric

acid and re-oxidation of the salts of iron by nitric acid it gave the following results:—In a preliminary experiment in duplicate, 30.84 per cent Zn; in a second 31.32 and 31.48 per cent Zn—either an error of more than 7 per cent of the mineral or of nearly 29 per cent of the Zn present. The solutions, after the deposition of the precipitate, were of a very deep yellow colour. The concordance of the results obtained with this sample of ore, on the one hand with sulphite of sodium, and on the other with ferrocyanide and the addition of sulphite, shows that the influence of the nitrite can be entirely done away with by this latter reagent. We, however, made a special experiment to prove it.

We used a 5 normal solution of nitrite of soda (6.9 grms. per litre) and a normal solution of sulphite of soda in the proportions given below.

The solutions were prepared as follows:—To 30 c.c. of $\frac{1}{2}$ normal zincic chloride we added the sulphite of soda, the nitrite diluted to 100 c.c. with water, 10 c.c. of 20 per cent chloride of ammonium, 10 c.c. of 5 normal hydrochloric acid, and 35 c.c. of ferrocyanide of potassium approximately $\frac{1}{2}$ normal for zinc. After standing not less than twenty minutes, we titrated back with the zincic solution.

	NaNO_2 $\frac{1}{2}$ normal.	Na_2SO_3 Normal.	ZnCl_2 $\frac{1}{2}$ normal.
1. . . .	—	0.5 c.c.	3.94 c.c.
2. . . .	1 c.c.	1.0	3.93
3. . . .	2	2.0	3.90
4. . . .	5	3.0	3.88
5. . . .	10	5.0	3.90

The use of sulphite thus neutralises the action of the nitrite absolutely; the final solution is quite colourless.

Influence of Manganese.—Manganese, which is sometimes found, though in small quantities only, in zinc ores, requires special care in its elimination. It has been thought that in acid solutions it was not precipitated by ferrocyanide, and was therefore without influence.

A quantitative experiment is sufficient to show that in order to prevent the precipitation of manganous ferrocyanide there must be present such a proportion acid as to seriously influence the precipitation of the zinc itself. However, we made a definite experiment. We used 20 c.c. of $\frac{1}{2}$ normal ZnCl_2 , 50 c.c. of $\frac{1}{2}$ normal ferrocyanide, 50 c.c. of 20 per cent AmCl , 10 c.c. of 5 normal HCl, and 100 c.c. of water, and digested for fifteen minutes.

1. Without manganese		ZnCl_2 back	4.50 c.c.
2. With 0.0275 grm. manganese	" "	" "	2.27 "

The manganese thus acted on the ferrocyanide the same as 2.23 c.c. of $\frac{1}{2}$ normal ZnCl_2 . The quantity of manganese added was equal to 2 c.c. of $\frac{1}{2}$ normal solution, and one sees that this metal requires for its precipitation at least as much ferrocyanide as zinc does. Manganese must therefore be entirely eliminated before titration.

(To be continued).

THE SOLENOID ELECTRO-MAGNET.

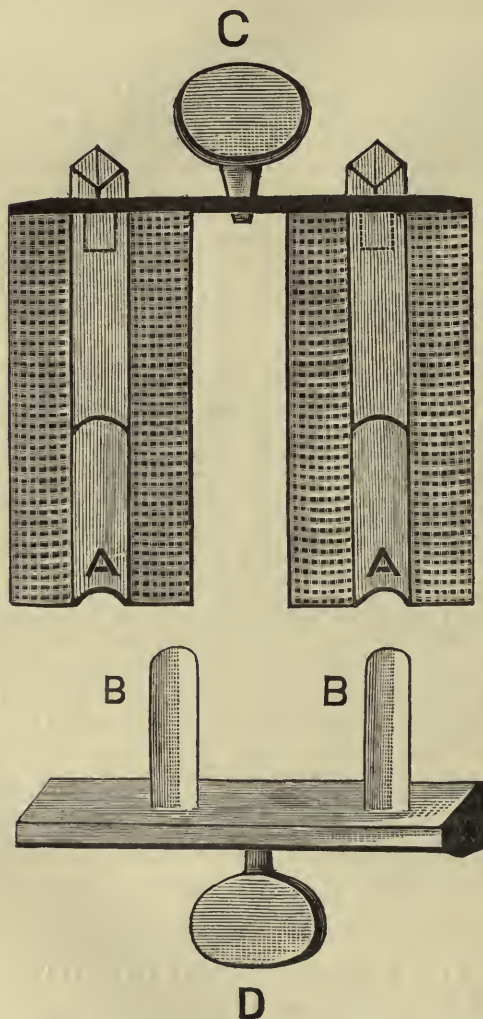
By H. N. WARREN, Principal, Liverpool Research Laboratory.

THIS powerful type of electro-magnets, which after a lengthy and exhaustive research, has just been perfected at the Research Laboratory, differs from all other magnets in the construction of the iron core; they being intended for either supporting great weights in general, or the accommodation of spherical bodies, for which reception a hollow cavity is cut from each extremity, the section of each cavity representing a semicircle.

For the construction of the iron core, iron oxides, free from sulphides, silicates, and carbonaceous matter, were selected, and reduced in an atmosphere of hydrogen,

puddled through a hot-blast Siemens furnace, and drawn into bars; the bars are next imbedded in quicklime, brought to a full white heat, and allowed to cool in that substance. After cooling, each limb before yoking is drilled to within half an inch of its circumference and one-quarter of its total length (if the bar be less than half inch from starting, a corresponding allowance has naturally to be made).

The following diagram illustrates the section of a magnet with its accompanying keeper, the separate parts being further described in the specified table. The magnet was constructed to suspend a weight of 10 tons, and required to be excited by a current obtainable from 25 boron carbon cells only.



From the above diagram it will be readily observed that when the keeper is applied with its two iron projections, B B, which are constructed of such dimensions as to exactly coincide with the openings A A; not only is the magnetic power fully utilised, but any side-slipping of the keeper is absolutely impossible. All the magnets thus described were wound with No. 14 double cotton-covered wire, the winding afterwards being covered with paraffined insulation, wound with thread, and varnished with best elecro shellac varnish: they are at present doing excellent service in researches on dimagnetism and polarisation of light.

The subjoined table will better explain a few of the more important magnets thus constructed, and giving approximate quantities of their component parts:—

Length and diameter of core.	Weight of primary.	Voltage required.	Weight supported.
2 inches $\times$ $\frac{1}{4}$	4 ozs.	6	8 lbs.
6 " $\times$ $\frac{1}{4}$	1 lb.	10	80 "
28 " $\times$ 2	9 lbs.	50	5 cwts.
28 " $\times$ 4	100 "	50	2 tons.
36 " $\times$ 4 $\frac{1}{2}$	112 "	50	10 "

18, Albion Street, Everton, Liverpool.
Liverpool Research Laboratory,

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING JUNE 30TH, 1897.

By WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolitan Water Act, 1871.

London, July 10th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 168 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from June 1st to June 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in a previous report.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 168 samples examined all were found to be clear, bright, and well filtered.

The rainfall at Oxford during June was 1·7 inches; the average for the last 30 years is 2·68 inches; this leaves a deficiency of 0·98 inch on the month, making an actual excess of 0·06 inch for the year, on a fall of 11·37 inches.

The results of our bacteriological examinations of 230 samples are recorded in the following table; we have also examined and reported on 34 other samples taken at various places, such as stand-pipes, and different filter-beds:—

	Microbes per c.c.
Thames water, unfiltered (mean of 24 samples)	73893
Thames water, from the clear water wells of five Thames-derived supplies (mean of 112 samples)	74
Ditto ditto highest	558
Ditto ditto lowest	6
New River, unfiltered (mean of 24 samples)	2005
New River, filtered (mean of 23 samples)	69
River Lea, unfiltered (mean of 24 samples)	66822
River Lea, from the clear water well of the East London Water Company (mean of 23 samples)	69

The remarks made last month as to the impossibility of comparing microbial estimations on the same water by different experimenters unless the samples were taken at the same time and place, and the subsequent processes

of treating the samples for plate cultivation of bacteria were absolutely identical, are strikingly borne out by a report recently addressed to the London County Council by their chemist, where stress is laid on a great discrepancy between the numbers given by Sir Edward Frankland and ourselves.

A discussion of differences of this kind will only be possible when all the analysts adopt the same method of bacteriological cultivation.

Samples of raw river water show continual variations according to the season, atmospheric conditions, and rainfall. For instance, on June 8th we found in the Thames, 270,320 microbes per c.c., and on the 9th 9920. On the 10th we obtained 982,080, and on the 11th 58,640. In the river Lea on June 14th we found over 1,500,000 microbes per c.c.: on the 16th, 240; on the 17th, 120; and on the 18th, 9920.

We take several samples daily and give the averages. Other chemists taking samples once a week or once a month could not, unless by mere chance, hit upon the averages we record.

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

NOTES ON LUCIUM.

By WALDRON SHAPLEIGH.

In May of last year I secured several samples of the so-called "lucium" which had been prepared by Dr. Leon Schützenberger, of Paris, and at the same time saw a report in which this chemist gave very fully the methods which he used in preparing it, and his conclusions regarding the same, also samples of the monazite sand used.

The methods he employed follow very closely those set forth in the patent of Mr. P. Barrière.

The monazite sand was a low-grade North Carolina sand, containing minerals rich in titanium, zirconium, and yttrite earths, such as menaccanite, rutile, zircon crystals, samarskite, euxenite, and xenotime, and it is probable that from the last three minerals the "lucium" is obtained.

On experimenting with a solution of the lucium nitrate the ignited ash or residue I found to be of a dirty grey colour, and nearly entirely soluble in hydrochloric and nitric acids even when diluted. This solution was clear and pink, and gave the absorption bands of erbium. As the time recommended by Mr. Barrière for digesting with the potassium sulphate for the separation of the cerite group seemed to be rather short, I saturated the solution with potassium sulphate in the cold and allowed it to stand forty-eight hours, thereby obtaining a precipitate consisting of the cerite group, and in the solution the yttrite group. Analysis showed:—

Yttrite group oxides	93.98
Lanthanum, cerium, didymium oxides ..	3.74
Thorium oxide	1.07
Titanium, zirconium, potassium, sodium oxides, and sulphuric acid, not separated	1.21
	100.00

The oxides of the yttrite group after this thorough purification by potassium sulphate were of a light buff or yellow colour, fully and completely soluble in dilute acids. Sodium hyposulphite did not give a precipitation in a fairly concentrated solution, nor was there any difficulty in making a partial separation of the erbium nitrate in the fused nitrates by Bahr and Bunsen's method. Although the quantity at my disposal was small, I obtained the erbium

nitrate crystals and showed the possibility of this separation.

Regarding the precipitation with sodium hyposulphite, on which great stress is laid as a reaction, showing "lucium" to be a new element, if a solution of yttrite sulphates in a concentrated solution of potassium sulphate made by cold digestion is heated, a copious precipitation will ensue. Though not a perfect separation, yet advantage can be taken of this reaction in separating yttrium from erbium, and this reaction should be borne in mind when separating the cerite from the yttrite group, as some text-books recommend using hot saturated solutions. By doing so, some of the yttrium will be precipitated with the cerite group.

This action of heat on the double sulphate solution of the yttrite group, if it does form similar salts to the cerite group, I have not met with in the text-books. Therefore, inasmuch as Schützenberger and Barrière added the sodium hyposulphite to the concentrated solution, and then heated the resulted mixture, I believe the precipitation was due to the above action of heat rather than to a precipitation by the sodium hyposulphite. In a 10 per cent solution of the yttrite group, oxides, I obtained but a slight precipitation with sodium hyposulphite, which, on resolution and treatment, did not again precipitate.

Barrière's methods introduce titanium and zirconium, both of which are exceedingly difficult to separate afterwards.

To obtain the pink colour of the erbium oxide it must be exceedingly pure. One can easily be misled by the colour of these rare oxides when in combination. Some when alone are white, yet when combined are of a dark colour, even brown.

In order to obtain a larger sample of "lucium" to work with, I took 1 kilo. of an average of several hundred samples of North Carolina monazite sand, carefully following Barrière's method, making, however, each separation thoroughly, and failed to obtain any earth answering to the reactions of "lucium," only getting as a result less than 1 per cent of the mixed oxides of the yttrite group of a buff colour and very freely soluble in dilute acids.

Since making these experiments much work has been done by Crookes and others to prove that "lucium" is not entitled to a place in the list of elements, but is impure yttrium.—*Journal of the Franklin Institute*, July, 1897.

BEHAVIOUR OF CHLORAL HYDRATE WITH AMMONIUM SULPHIDE.

By JOSEPH LESINSKY and CHARLES GUNDLICH.

WHILE studying the reactions of chloral hydrate, we observed that, when dissolved in water and mixed with a little ammonium sulphide, it threw down a precipitate only after a certain time had elapsed. This precipitate separated out almost instantaneously after a certain number of seconds had been counted. Upon further experiment we found that, if the concentrations are the same, the precipitate will appear, every time the experiment is tried, in exactly the same number of seconds, provided the temperature is the same.

At first we had considerable difficulty in repeating the experiment, as we had prepared a fresh quantity of ammonium sulphide. It was found, however, that this did not react with the chloral hydrate, even after standing a long time. We were more successful on using a well-saturated solution of ammonium sulphide, and especially a solution that had been standing for several days,—in other words, a solution of the polysulphides.

To show this reaction, the following conditions were observed:—

Two grms. of chloral hydrate were dissolved in 25 c.c. of water, 10 c.c. of this solution run out of a burette into

a narrow beaker, and 5 c.c. of yellow ammonium sulphide poured in, not run in. The liquids must be mixed as quickly as possible. It is best to shake the mixture slightly, and allow it to stand on a piece of white paper, so that the sudden appearance of the precipitate can be seen from a distance in showing the experiment.

At different temperatures the reaction is retarded or hastened, the time necessary for the precipitate to form being inversely proportional to the temperature and concentration.

At 1° C. the separation took place in 44 seconds.	
" 20° C. " " 19 "	
" 45° C. " " 8 "	
" 65° C. " " 3 "	

For every 20° rise we have a gradual decrease in time.

The difference between 45° and 65° being 5 seconds.	
" " 45° and 20° " 10 "	
" " 20° and 1° " 20 "	

The best way to show this reaction is to start a metronome ticking, mix the two solutions quickly, and; while observing the mixture, count the number of seconds it takes before the precipitate appears. The exact nature of this reaction is not understood, especially as the resulting precipitate is evidently a mixture of sulphur and perhaps some sulphur compound of a mercaptan character, the odour being very characteristic. The precipitate was filtered off, and the solution, which was red, extracted with ether, the resulting oil emitting a very powerful odour, and, when diluted with water, suggesting the odour of walnuts. If the concentrations of the two substances are changed, the resulting precipitate will vary in appearance. In our first experiments the precipitate was of a pinkish colour, and the solution red; on heating a little on the water-bath, the precipitate became yellow and then brown, a remarkable succession of changes in colour being observed. In the above-mentioned experiments the precipitate was yellowish brown in colour, the solution itself being yellow, and on warming the precipitate the colour became almost black.

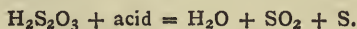
In order to obtain the pink precipitate it is advisable to use an excess of chloral hydrate, but it requires some experimenting to obtain the conditions favourable for the formation of the precipitate.

Perhaps these reactions may be of value in the examination of chloral hydrate as to its purity. It is our intention to study the same, and also to determine the nature of the precipitate and the cause of the "retarded precipitation."

There are only two such cases of retarded precipitation known to us, besides the one described by us, and, even in those cases, it has been impossible to ascertain the causes, although the reactions are not complicated, as in the case with the one described by us.

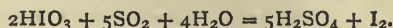
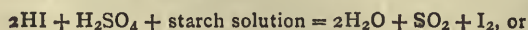
The reactions are as follows:—

1. The action of sodium thiosulphate upon hydrochloric acid:—



Here the sulphur separates out suddenly after a lapse of time.

2. The action of hydriodic acid with a starch solution when sulphuric acid is added:—



The last reaction is the most characteristic of the three, the iodine separating out so suddenly that most accurate results can be obtained in measuring the time. We have furthermore succeeded in procuring the same results by employing butyl chloral (croton chloral) instead of chloral hydrate, the resulting precipitate being of a beautiful lemon-yellow colour. This last reaction is not as striking

as the others, the precipitate requiring two or three seconds for complete separation.—*American Chemical Journal*, vol. xix., No. 7.

ACTION OF PHOSPHORUS PENTACHLORIDE ON ANILINE AND ITS SALTS.*

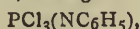
By J. ELLIOTT GILPIN.

THE constant occurrence of chlorophosphuret of nitrogen (PNCl_2)₃ as a by-product in the formation of orthosulphobenzoic acid from commercial saccharine directed attention in this laboratory (Chem. Labor., Johns Hopkins University) to this very interesting substance. Discovered by Liebig, it was studied by Liebig and Wöhler (*Ann. Chem.*, Liebig, xi., 139), Gladstone and Holmes (*Ann. Chem.*, Liebig, lxxvi., 74), Laurent, (*Comptes Rendus*, 1850, Sept. 9), Wichelhaus (*Ber. d. Chem. Ges.*, iii., 163), Hofmann (*Ber. d. Chem. Ges.*, xvi., 1910), and Stokes (*Amer. Chem. Journ.*, xvii., 275). Hofmann studied its action with aniline and paratoluidine, and obtained derivatives in which the chlorine had been replaced by residues of aniline and paratoluidine, the product with the aniline having the composition $\text{P}_3\text{N}_3(\text{NHC}_6\text{H}_5)_6$. Besson (*Compt. Rendus*, cxiv., 1264) has repeated the work done on this substance and obtained the compound PNCl_2 . He says the compound described by Gladstone as having the composition (PNCl_2)₃ and the melting-point 210° is probably a polymeric form of PNCl_2 ; but there is evidently some mistake here, as Gladstone found the melting-point to be 110° and not 210°, while according to Besson it is 114°. As the formation of the chlorophosphuret of nitrogen is due to the action of phosphorus pentachloride on ammonium chloride, the possibility was suggested of obtaining a derivative of it by using instead of ammonium chloride a substitution-product, aniline hydrochloride. Although the substance obtained in this action is not a derivative of the chlorophosphuret of nitrogen, still a comparison of the two forms an interesting study. They show the tendency of phosphorus and nitrogen to combine sometimes in a very stable form, as in the chlorophosphuret of nitrogen and a substance obtained in this investigation, which will be discussed later. The former can be heated with concentrated acids and alkalis without undergoing change; but the trichlorophosphanil, obtained in this work, is very unstable, decomposing with nearly all reagents, and even slowly when exposed to moisture.

Up to the time when this investigation was begun the study of the action of aniline on the chlorides of phosphorus had not led to satisfactory results, and the action with the trichloride only had been studied. By the action of aniline on phosphorus trichloride, Tait (*Zeitsch. Chem.*, 1865, 648) obtained a compound to which he gave the formula $\text{P}(\text{NH}_2\text{C}_6\text{H}_5\text{Cl})_3$. Jackson and Menke (*Amer. Chem. Journ.*, vi., 89) repeated this work, but were unable to obtain the product described by Tait. They obtained a product which they thought had the composition $\text{PCl}(\text{NHC}_6\text{H}_5)_2$, but were unable to isolate it. By dissolving it in alcohol and precipitating with water they obtained the compound $\text{P}(\text{OH})(\text{NHC}_6\text{H}_5)_2$, which they considered to be its hydroxyl derivative. Weyer ("Dissertation," Bonn, 1891) studied the action of aniline on trichloride of arsenic, and obtained two definite products by the successive substitution of aniline residues for the chlorine atoms. These were represented by the formulæ $\text{AsCl}_2(\text{NHC}_6\text{H}_5)$ and $\text{AsCl}(\text{NHC}_6\text{H}_5)_2$. The third step he was unable to take. In the present investigation, in which the action of phosphorus pentachloride on aniline and its salts is under investigation, four definite substances have been isolated. They are formed by the substitution of residues of aniline for one or more chlorine

* *American Chemical Journal*, vol. xix., No. 5.

atoms of the phosphorus pentachloride, one being obtained by the action of phosphorus pentachloride on aniline hydrochloride, and three by the action on aniline. The trichlorophosphanil, having the composition—



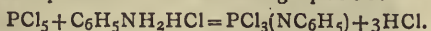
which was obtained from the aniline hydrochloride, shows great similarity to the first derivative obtained by Weyer. They are both very sensitive to moisture, and decompose with water and alcohol in a similar manner. The other substances were more stable, and were obtained from their solutions in alcohol, in the form of well-defined crystals.

During the progress of this investigation an article by Michaelis and Schroeter (*Ber. d. Chem. Ges.*, xxvii., 490) has appeared, in which a substance, formed by the action of aniline on phosphorus trichloride, of the composition $\text{C}_6\text{H}_5\text{N}-\text{PCl}_2$, is described. This substance bears the same relation to phosphorus trichloride as the trichlorophosphanil, described in this paper, does to phosphorus pentachloride.

Action of Phosphorus Pentachloride on Aniline Hydrochloride—Trichlorophosphanil, $\text{PCl}_3(\text{NC}_6\text{H}_5)$

After considerable experimenting, the following method was adopted as the best for the preparation of this substance:—Phosphorus pentachloride and perfectly dry aniline hydrochloride are mixed in molecular quantities, not more than 10 grms. of the aniline hydrochloride being used at a time. The intimately mixed compounds are placed in a distilling-bulb, and connected with a condenser. A tube at the lower end of the condenser passes into a vessel of water, to absorb the hydrochloric acid formed and show the rate of the action. The bulb is half immersed in a sulphuric-acid bath. Before the temperature at which the trichlorophosphanil is formed is reached, a small quantity of phosphorus pentachloride sublimes in the upper part of the neck. If the temperature is kept at 170° for six to eight hours, the trichlorophosphanil condenses on the cool part of the bulb, and sometimes in the condenser, as a white coating, covered on the inside with nodules. After the evolution of hydrochloric acid has ceased, the material can be obtained in a very pure condition, if the part in the neck of the bulb is discarded. After being powdered, it is placed in a desiccator over caustic potash to remove the hydrochloric acid. It must be kept perfectly dry, and cannot be purified by crystallisation, as water, alcohol, ether, acetone, and other solvents decompose it. Even when re-sublimed some decomposition takes place. In every case there was a small quantity of a dark, green, sticky material left in the bulb after the action was over. This may have been due to impurities in the phosphorus pentachloride, as the ordinary commercial article was used. When heated with water this green material acts quite violently at first, in consequence no doubt of the presence of a small excess of phosphorus pentachloride, and a tar-like mass is formed, which is unacted on by water, alkali, and dilute acid, and solidifies on cooling. It is soluble in alcohol, with a green colour; so some was dissolved in alcohol and precipitated by addition of water, filtered, and washed. This was repeated several times, and the green powder obtained was analysed; but different preparations varied in composition, and it was evidently not a single definite compound.

The formation of the trichlorophosphanil probably takes place as represented in the following equation:—



Concentrated sulphuric and nitric acids both decompose it, the former with the evolution of hydrochloric acid. Alcohol and ether act on it with the evolution of hydrochloric acid and the generation of considerable heat. Sodium hydroxide acts on it to a slight extent, and changes it to a hard brittle mass. When boiled with water it is decomposed into aniline, hydrochloric and

phosphoric acids, so that the chlorine and phosphorus can be determined in the same specimen, the chlorine as silver chloride, and the phosphorus as magnesium pyrophosphate. To be sure that none of the hydrochloric acid was lost in boiling, several determinations were made by the Carius method, but they agreed with those made in the other way.

The following are the results of the analyses:—

Preparation I.

0.3662 grm. of the substance gave 0.6754 grm. AgCl.
0.2848 grm. of the substance gave 0.5226 grm. AgCl.
0.2560 grm. of the substance gave 0.4672 grm. AgCl.
0.3662 grm. of the substance gave 0.1788 grm. $\text{Mg}_2\text{P}_2\text{O}_7$.
0.2848 grm. of the substance gave 0.1367 grm. $\text{Mg}_2\text{P}_2\text{O}_7$.
0.2560 grm. of the substance gave 0.1242 grm. $\text{Mg}_2\text{P}_2\text{O}_7$.
0.2755 grm. of the substance gave 0.0609 grm. H_2O and 0.3196 grm. CO_2 .
0.4078 grm. of the substance gave 22 c.c. N at 16° and 772.5 m.m.

Preparation II.

0.2568 grm. of the substance gave 0.4635 grm. AgCl.
0.3535 grm. of the substance gave 0.6354 grm. AgCl.
0.3535 grm. of the substance gave 0.1695 grm. $\text{Mg}_2\text{P}_2\text{O}_7$.
0.4237 grm. of the substance gave 23 c.c. N at 11.5° and 762 m.m.
0.3698 grm. of the substance gave 0.0765 grm. H_2O and 0.4081 grm. CO_2 .
0.3646 grm. of the substance gave 0.0748 grm. H_2O and 0.4078 grm. CO_2 .

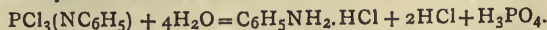
Calculated for $\text{PCl}_3(\text{NC}_6\text{H}_5)$.	Preparation I.		Found.		Preparation II.	
C	31.55	—	31.63	—	30.09	—
H	2.19	—	2.45	—	2.29	—
Cl	46.53	45.59	45.36	45.11	44.43	—
P	13.58	13.64	13.41	13.56	—	13.40
N	6.17	—	6.38	—	—	6.47

While these results leave much to be desired, they point clearly to the formula given, and are fairly satisfactory considering the instability of the substance.

The sulphate and nitrate of aniline were also treated with phosphorus pentachloride, but they did not act like the hydrochloride. The action of the nitrate was very violent, taking place as soon as they were mixed, without the aid of heat. The whole mass was carbonised in a short time. Paratoluidine hydrochloride was also treated with phosphorus pentachloride, but no volatile product was formed.

Decomposition of Trichlorophosphanil with Water.

When trichlorophosphanil is heated with water it first melts and forms an oil which is decomposed by further boiling. The decomposition gives rise to the formation of aniline, and hydrochloric and phosphoric acids, and can be represented thus:—



The crystals which separated out, after decomposing with water and evaporating, were re-crystallised and analysed. The results of the analyses showed it to be aniline hydrochloride. Another portion was decomposed with water, an excess of barium hydroxide added to liberate the aniline, and this then distilled off. After filtering off the excess of the hydroxide, the presence of barium chloride was proved, showing that free hydrochloric acid had been present. The barium was precipitated with sulphuric acid and the phosphoric acid obtained by evaporating the solution.

Decomposition of Trichlorophosphanil with Sulphuric Acid.

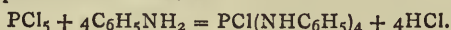
Trichlorophosphanil dissolves in warm concentrated sulphuric acid, with evolution of hydrochloric acid gas. An equal volume of water is added to this, and the liquid is allowed to stand, when a substance crystallised out which

was found to be sulphanilic acid. Several of its salts were made and analysed.

Action of Phosphorus Pentachloride on Aniline. Chlorophostetranilide, $\text{PCl}(\text{NHC}_6\text{H}_5)_4$.

When phosphorus pentachloride is slowly added to aniline and the mass constantly stirred, there is immediate action and hydrochloric acid is evolved. The whole mass becomes hot, and after all the aniline has been used up, and further addition of phosphorus pentachloride causes no action, it solidifies. This mass, when cool, is broken up and heated with water to extract any aniline hydrochloride and phosphorus pentachloride present, then filtered, washed, and dried. This product, which is a light yellow powder, is insoluble in water, but slightly soluble in alcohol. It consists largely of one substance, but several others are present in small quantities.

If the dried mass is shaken with two separate quantities of cold alcohol, and the liquid, after being filtered, is allowed to evaporate slowly, besides some needle-shaped crystals there will be found some well-defined monoclinic crystals, which grow to a considerable size. These have to be separated from the other product mechanically. If the solution from which these have been obtained is filtered and allowed to evaporate still further, in some cases, tufts of fine, radiating, silky needles separate out. Both these and the large crystals will be discussed later. After treating several times with cold alcohol, the original material is treated repeatedly with small quantities of hot alcohol, until on evaporation only one substance separates out from the solution. By this process a definite substance is obtained in pure condition. This is slightly soluble in alcohol and insoluble in water. When crystallised from alcohol it usually consists of long irregular-shaped crystals; but when the evaporation is very slow, and absolute alcohol is used, monoclinic crystals, with well-defined basal planes and fundamental prisms, are formed. The composition of this substance is represented by the formula $\text{PCl}(\text{NHC}_6\text{H}_5)_4$, and its mode of formation is expressed as follows:—



It is not decomposed when boiled with water, concentrated alkali, or hydrochloric acid; but when heated with water in a sealed tube to 180° for several hours, aniline, aniline hydrochloride, and phosphoric acid are formed. This substance is characterised by its great insolubility. It is practically insoluble in ethyl ether, acetone, benzene, ligroin, and amyl alcohol, but slightly soluble in ethyl alcohol. When heated, a volatile product, which proved to be a mixture of aniline, aniline hydrochloride, and some diphenylamine hydrochloride, formed by the action of aniline on aniline hydrochloride, was formed, and an amorphous black substance remained. The great stability of this substance is shown by the fact that only a small amount was decomposed when it was heated in a porcelain tube, in a current of oxygen, over the blast-lamp for several hours.

A qualitative analysis of this substance showed that it contains phosphorus, nitrogen, and carbon. The black stable residue is very similar to a product described by Rose (*Ann. der. Phys. Pogg.*, xxviii., 529; *Ann. Chem.*, Liebig, xi., 130). By the action of phosphorus trichloride on ammonia he obtained a product in which he gave the formula $\text{PCl}_3 + 5\text{NH}_3$. When this was heated, ammonium phosphate and ammonium chloride were formed, and a white substance, phospham, PN_2H , was left behind. A comparison of this and the black residue is worthy of notice. Neither is soluble in water or dilute acids. They are only partly decomposed by boiling with fuming nitric acid for some time. Concentrated sulphuric acid attacks the phospham, but acid potassium sulphate is necessary to decompose the black residue. Neither is acted on when heated in a stream of chlorine. They are decomposed by fused alkali, and also with generation of phosphorus or a compound of phosphorus and hydrogen, when heated to

redness in a current of hydrogen. The black residue may also contain a small amount of hydrogen; but this cannot be considered as certain, for the decomposition of the substance is accomplished with so great difficulty that the sources of error introduced may account for the presence of the hydrogen.

The formation of this compound prevented the determination of the carbon and hydrogen by the usual method in the case of all the compounds studied, except that of the trichlorophosphanil, which is volatile. In some cases, when the substances were heated in platinum boats, a transparent film was formed about the small particles of carbon, preventing their complete combustion. The method finally adopted for the determination of carbon, and the only one by which satisfactory results could be obtained, was that described by Fritsch (*Ann. Chem.*, Liebig, ccxciv., 79), in which the substance was oxidised by potassium chromate and sulphuric acid.

The nitrogen was determined both by the absolute and by the Kjeldahl methods. The chlorine and phosphorus were determined as silver chloride and magnesium pyrophosphate respectively, the substance being decomposed by heating it with fuming nitric acid in a sealed tube to 180° for several hours.

(To be continued.)

ON THE DOUBLE FLUORIDES OF ZIRCONIUM WITH LITHIUM, SODIUM, AND THALLIUM.

By H. L. WELLS and H. W. FOOTE.

In a previous article (*Amer. Journ. Science*, IV., i., 18) we have described the caesium-zirconium fluorides, and upon comparing these with the corresponding ammonium and potassium salts, which had been previously described by Marignac (*Am. Chim. Phys.*, ix., 257) it was observed that the types of salts formed varied with the molecular weights of the alkaline fluorides in an interesting manner. The fluorides of smaller molecular weight gave types with a larger relative number of these molecules, while the fluorides of higher molecular weights combined with more zirconium fluoride than the others. This relation is made clear in the following table, which was given in the previous article referred to:—

3 : 1 Type.	2 : 1 Type.	1 : 1 Type.	2 : 3 Type.
$3\text{NH}_4\text{F} \cdot \text{ZrF}_4$	$2\text{NH}_4\text{F} \cdot \text{ZrF}_4$	—	—
$3\text{KF} \cdot \text{ZrF}_4$	$2\text{KF} \cdot \text{ZrF}_4$	$\text{KF} \cdot \text{ZrF}_4$	—
—	$2\text{CsF} \cdot \text{ZrF}_4$	$\text{CsF} \cdot \text{ZrF}_4$	$2\text{CsF} \cdot 3\text{ZrF}_4 \cdot 2\text{H}_2\text{O}$

The present investigation was undertaken with the view, in the first place, of testing the apparent rule with lithium fluoride, which has a lower molecular weight than the fluorides previously experimented upon. Our expectations were realised by the preparation of the salt $4\text{LiF} \cdot \text{ZrF}_4 \cdot \frac{2}{3}\text{H}_2\text{O}$. The salt $2\text{LiF} \cdot \text{ZrF}_4$ was also obtained, but, in spite of a careful search, no intermediate 3 : 1 salt could be discovered. The following table, giving the lithium, potassium, and caesium salts, shows a perfectly symmetrical gradation in types according to the atomic weights of the alkali metals, except that the intermediate lithium salt is missing:—

Type.	Lithium salts.	Potassium salts. (Marignac).	Caesium salts.
4 : 1	$4\text{LiF} \cdot \text{ZrF}_4 \cdot \frac{2}{3}\text{H}_2\text{O}$	—	—
3 : 1	—	$3\text{KF} \cdot \text{ZrF}_4$	—
2 : 1	$2\text{LiF} \cdot \text{ZrF}_4$	$2\text{KF} \cdot \text{ZrF}_4$	$2\text{CsF} \cdot \text{ZrF}_4$
1 : 1	—	$\text{KF} \cdot \text{ZrF}_4 \cdot \text{H}_2\text{O}$	$\text{CsF} \cdot \text{ZrF}_4 \cdot \text{H}_2\text{O}$
2 : 3	—	—	$2\text{CsF} \cdot 3\text{ZrF}_4 \cdot 2\text{H}_2\text{O}$

Marignac's two ammonium salts, 3 : 1 and 2 : 1, also enter the series symmetrically.

We have investigated also the thallous zirconium

fluorides, since the high atomic weight of thallium led us to expect that it would possibly yield a series of salts symmetrical with those of the alkali metals with a still higher ratio of zirconium than was the case with caesium. Such was not the case, however. The salts discovered were:— $3\text{TiF}_2.\text{ZrF}_4$, $5\text{TiF}_3.\text{ZrF}_4.\text{H}_2\text{O}$, $\text{TiF}.\text{ZrF}_4$, and $\text{TiF}.\text{ZrF}_4.\text{H}_2\text{O}$. Two of these three types of thallose salts correspond to types of alkali-metal salts; while one type, the 5:3, is a new one, but the series is not symmetrical with the others according to the atomic weights.

Since Marignac had described but one sodium-zirconium fluoride, $5\text{NaF}.2\text{ZrF}_4$, and since this differs from all other alkaline zirconium fluorides, we have undertaken a new investigation of the sodium salts. As a result, we have fully confirmed Marignac's results as to the 5:2 salt, which is the one most readily obtained, and we have succeeded in preparing a new salt, $2\text{NaF}.\text{ZrF}_4$, which corresponds to the most usual type of double halogen salts of tetravalent elements. It is evident, however, that the sodium salts, like those of thallium, do not form a symmetrical series with the others.

The following table gives a list of the sodium and thallium salts and shows the positions, "X," of the other compounds prepared by Marignac and ourselves:—

Type.	Lithium salts.	Ammonium salts. (Marignac).	Sodium salts.	Potassium salts. (Marignac).	Cesium salts.	Thallium salts.
4:1	X	—	—	—	—	—
3:1	—	X	—	X	—	$3\text{TiF}_2.\text{ZrF}_4$
5:2	—	—	$5\text{NaF}.2\text{ZrF}_4$	—	—	—
2:1	X	X	$2\text{NaF}.\text{ZrF}_4$	X	X	—
5:3	—	—	—	—	—	$5\text{TiF}_3.\text{ZrF}_4.\text{H}_2\text{O}$
1:1	—	—	—	X	X	$\text{TiF}.\text{ZrF}_4$
2:3	—	—	—	—	X	$\text{TiF}.\text{ZrF}_4.\text{H}_2\text{O}$

While our investigation has shown that the rule for the variation of the types with the atomic weights applies only partially to the zirconium double fluorides, we have shown at least that the variety of types is remarkable, and it is also noticeable that the ratios are nearly the simplest that can exist in such number between the extreme limits 4:1 and 2:3.

Preparation.—Thallium fluoride was prepared by dissolving the metal in sulphuric acid, adding an excess of baryta water, filtering, and passing carbonic acid into the hot solution. The filtrate from this precipitation was evaporated and treated with hydrofluoric acid in excess. The salts were prepared by mixing the acid solutions of the fluorides in varying proportions, evaporating, and cooling to crystallisation. The salts were then removed and pressed between filter papers till dry. In all cases they were stable in the air.

Method of Analysis.—Zirconium and the alkalis were determined by evaporating the salt with sulphuric acid to drive off hydrofluoric acid, precipitating zirconium hydroxide with ammonia and weighing ZrO_2 . The filtrate was evaporated to dryness and the alkali determined as sulphate, either by igniting with ammonium carbonate or heating in a current of air containing ammonia. When thallium was present, the fluoride was dissolved in water, a little sulphurous acid added to make sure that the thallium was all in the univalent condition, and the zirconium precipitated with ammonia. The precipitate needed to be very thoroughly washed. The filtrate was evaporated nearly or quite to dryness to remove free ammonia, diluted to a volume of about 100 c.c., heated to boiling, and potassium iodide added in excess to precipitate thallium iodide. This was collected on a Gooch crucible, washed with 80 per cent alcohol, dried at 100°C ., and weighed.

Fluorine was determined by the ordinary calcium fluoride method after precipitating zirconium with ammonia and removing ammonium salts by evaporation with sodium carbonate. Water was determined by heating the salt in a combustion tube behind a layer of dry sodium carbonate and collecting the water in a calcium chloride tube.

Salts of Lithium.

$2\text{LiF}.\text{ZrF}_4$.—This salt forms when from 0.7 gm. to 2 grms. of lithium fluoride are added to 20 grms. of zirconium fluoride. The crystals are hexagonal, showing prism and pyramid, and rarely a basal plane. In appearance they are very much like crystals of quartz from Herkimer Co., N.Y., but they are very small. On re-crystallising, the 4:1 salt was formed.

Separate crops were analysed with the following results:—

	I.	II.	Calculated for Li_2ZrF_6 .
Li. . . .	6.03	6.39	6.42
Zr	41.81	41.64	41.28
F	—	51.62	52.30

$4\text{LiF}.\text{ZrF}_4.3\text{H}_2\text{O}$.—This was the most unsatisfactory salt obtained, though it seems undoubtedly to establish the 4:1 type. As lithium fluoride is very insoluble, only a comparatively small amount could be dissolved in zirconium fluoride, and apparently we could not go far enough toward the lithium end to get the salt in pure condition. It formed in a crust ordinarily, and the crystals were very small. Under the microscope, no mixture with another salt could be found in the crops analysed. Once, however, it was obtained mixed with the 2:1 salt, as seen under the microscope, showing there could probably be no intermediate salt. Various conditions were tried, and crops were obtained from both hot and cold solutions. It forms when 5 to 7 grms. of lithium fluoride are mixed with 20 grms. of zirconium fluoride. On re-crystallising, lithium fluoride is precipitated.

Following are the results of the analyses:—

	Li.	Zr.	H_2O .	F.
I.	9.54	33.14	4.83	—
II.	—	—	4.93	—
III.	9.79	33.30	4.35	53.16
IV.	—	33.23	—	—
V.	—	33.02	—	—
Calculated for $\text{Li}_4\text{ZrF}_8.3\text{H}_2\text{O}$	9.93	31.91	4.26	53.90

Salts of Sodium.

$2\text{NaF}.\text{ZrF}_4$.—This salt crystallises in very minute crystals of hexagonal outline, coming down in a crust when from one to two parts of sodium fluoride are added to fourteen parts of zirconium fluoride. The salt does not re-crystallise. The following results were obtained from separate crops. The water was probably mechanically included.

	I.	II.	Calculated for Na_2ZrF_6 .
Na	18.66	18.41	18.40
Zr	34.78	36.21	36.00
H_2O	1.96	0.50	—
F (by difference). . . .	44.60	44.88	45.60

$5\text{NaF}.2\text{ZrF}_4$.—Marignac has previously described this salt, which comes down under wide conditions in very good crystals and re-crystallises easily. Prof. L. V. Pirsson has kindly examined the crystals and made the following report:—

"The crystals show good sharp forms, but are very small. They appear distinctly orthorhombic in habit, consisting in the main of rather stout prisms, made up of two prismatic planes, m and m' , and terminated by a rather steep brachydome. In another habit, which is rarer, the front pinacoid, a , is broadly developed, while the prisms are very small; this type also shows at times

a pyramid, p . The plane of the optic axes lies in the base and $a=c$, $b=a$, $c=b$. The optic angle is large, and it could not be told whether a or b was the acute diædrix. The double refraction is very low. The crystals in their form strongly recall the figures of chrysolite (olivine) shown in the mineralogies."

The analyses gave the following results from different crops:—

	I.	II.	Calculated for $\text{Na}_2\text{Zr}_2\text{F}_{12}$.
Na	21'15	21'09	21'23
Zr	33'63	33'55	33'22
F (by difference).	45'22	45'36	45'57

Salts of Thallium.

$\text{TlF} \cdot \text{ZrF}_4 \cdot \text{H}_2\text{O}$ and $\text{TlF} \cdot \text{ZrF}_4$.—These salts crystallise in somewhat concentrated solutions when one part of thallium fluoride is mixed with three or four parts of zirconium fluoride. The analyses invariably show an excess of zirconium fluoride. The hydrous salt crystallises in needles, if the solution be cooled before precipitation occurs. If the solution is evaporated until crystals begin to form and then cooled, the anhydrous salt deposits in minute square plates. The salt gives the 5:3 type on re-crystallising. The following results were obtained:—

	I.	II.	Calculated for $\text{TlZrF}_6 \cdot \text{H}_2\text{O}$.
Tl	48'43	47'91	50'05
Zr	22'93	23'16	22'15
F	—	23'17	23'37
H_2O	3'89	4'80	4'43

	I.	II.	Calculated for TlZrF_6 .
Tl	50'16	49'91	52'37
Zr	23'86	24'08	23'17
F	—	24'32	24'46

$5\text{TlF} \cdot 3\text{ZrF}_4 \cdot \text{H}_2\text{O}$.—This salt crystallises in needles when from 1 to $3\frac{1}{2}$ parts of thallium fluoride are added to 1 part of zirconium fluoride. When about 4 parts of thallium fluoride are added, the same salt crystallises in a different habit, forming prisms of hexagonal outline which under the microscope are seen to be twinned, resembling in this respect the hexagonal-shaped crystals of aragonite. On re-crystallising, both habits give the needle-shaped crystals.

The following analyses were made of the two kinds of crystals. A rather large number of determinations was made on account of the existence of two different forms.

	Tl.	Zr.	H_2O .	F.
I... .. .	61'58	16'88	—	—
II... .. .	62'05	16'84	—	—
III... .. .	61'37	17'14	1'40	—
IV... .. .	61'58	16'88	1'17	19'31
V... .. .	61'74	17'04	1'42	—
VI... .. .	—	—	1'31	—
VII... .. .	62'91	16'42	—	—
Calculated for $\text{Tl}_5\text{Zr}_3\text{F}_{17} \cdot \text{H}_2\text{O}$	62'47	16'58	1'11	19'84

$3\text{TlF} \cdot \text{ZrF}_4$.—Crystals of this salt form in brilliant octahedra when one part of zirconium fluoride is added to from four to twenty parts of thallium fluoride. It is easily re-crystallised.

The following analyses were made:—

	I.	II.	Calculated for Tl_3ZrF_6 .
Tl	72'82	73'20	73'24
Zr	10'91	10'38	10'80
F	15'65	—	15'96

—*American Journal of Science*, iii., No. 18, June, 1897.

NOTICES OF BOOKS.

Journal of the Tokyo Chemical Society ("Tokyo Kwagakukwai"), Vol. xviii., No. 3, March, 1897.

Molecular Conductivity of Amido-sulphonic Acid.—J. Sakurai.—This paper, read before the Society and printed in Japanese characters, takes up about two-fifths of the number, and is illustrated with cuts of the apparatus used. The remaining pages are devoted to Abstracts of Chemical Papers published in other Journals and Miscellaneous Notes. The journal appears to be doing well, but, owing to unavoidable circumstances, we are unable to make any detailed observations on the contents.

Vol. xviii., No. 4, April, 1897.

This number contains an original paper entitled "Report of Experiments on the Manufacture of Japan Camphor (No. II.)," by M. Moriya, Rigakushi. The rest of the number is taken up with the Report on the general Proceedings of the Society, Abstracts of Chemical Papers published in other Journals, and Miscellaneous Notes.

The Agricultural Journal of the Cape of Good Hope. Vol. x., No. 11, May 27, 1897. Cape Town: Townshend, Taylor, and Snashall.

In this number we find an account of some interesting experiments on rinderpest, which is now ravaging South Africa; they were carried out by Dr. Kohlstock under the direction of Dr. Koch. He found that immunisation was produced by serum, but is of short duration, not longer than from ten to twenty days. Serum taken from healthy full-grown animals after mild rinderpest, which had been inoculated with 20 c.c. of virulent blood, without effect, to prove its immunity, gave the strongest serum. Twenty-four cattle were used for this experiment. The best serum was given by an animal which was first injected with gall, and then with rinderpest blood, before immunity was established, so that it suffered from a mild attack: this is the method recommended by Dr. Koch, and has been established by further experiments. The serum seems to be strongest when taken between the tenth and twentieth days—say the fifteenth.

One animal appeared to be naturally immune; it was an ox which had been destined to produce gall; but the introduction of 10, 20, and even 50 c.c. of rinderpest blood failed to produce any effect. Experiments are being made with serum from this animal. Two calves born on the station proved to be immune; they were both produced by cows which had previously been rendered immune. Dr. Kohlstock firmly believes that if gall inoculation is made in the manner indicated, rinderpest will soon be a thing of the past in South Africa: we sincerely hope this is the case.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 25, June 21, 1897.

Examination of some Spectra.—Lecoq de Boisbaudran.—MM. Eder and Valenta resume the critique of my spectrum of Au undertaken by Prof. Krüss. Demarçay has shown that the objections of Prof. Krüss are unfounded. Except the weak ray 433'8, the origin of which I do not presume to assert, but which I always see in the

non-condensed spark with the very concentrated solution of AuCl_3 . M. Demarçay has recognised all my metallic rays. It was, moreover, improbable that the strong ray Au 523.0 could be due to Pd, and that Au 406.4, the strongest of all with a short wire, was alien to gold. The pure gold of commerce would give an appreciable spectrum of palladium, and I did not fail to thoroughly purify my gold. Except 433.8, all my metallic rays have been seen by Eder and Valenta. The other rays (except, possibly, 592.5) are found in the spectrum of the flame of AuCl_3 . The weak ray 592.5 (A.) is difficult to see or to measure. I have obtained it formerly with AuCl_3 and electrodes of Pd or Pt. Having examined it again, I have found it slightly fainter and more cloudy than it is shown in my sketch. Measurements gave about 592.56 (A.). In the flame, the thick band 591.3 would mask 592.5 if this ray exists there. I see therefore always the faint ray 592.5 with AuCl_3 in the spark, but I cannot assert anything as to its origin. Eder and Valenta have a faint and indistinct ray at 592.143 (Rd.), 592.043 (A.). Can this be the same? If I read their text correctly, Eder and Valenta say that my band 560.1 does not belong to gold, no more than my ray 521.2. On referring to my sketches, we find 560.1 and 521.2 among the principal rays or bands volatilised in the flame. Eder and Valenta say that my ray 444.2 does not belong to gold. I have not this ray, but I have one at 443.77 (A.), 443.84 (Rd.), but it has been seen by Eder and Valenta at 443.787 (Rd.). I have not given the ray 434.5, I have only seen there the ray 433.81 (A.), 433.87 (Rd.). I have made new experiments on the subject of this ray 433.8. A series of measurements (difficult) has given 433.83 (A.), 433.89 (Rd.). In a vacuum tube the Cl has a dense cloudy ray, which I have measured at about 434.30 (A.). This seems to me rather remote from 443.83. Moreover, the aspects of the rays are very different. In the open air, between platinum points, the condensed spark gives a thick ray which I find at 434.75 (A.); this is far from 433.83. In the neighbourhood of 433.8 I only see with the condensed spark (taken between dry or moist poles) a faint ray, sensibly more refrangible than that seen with AuCl_3 . The ray AuCl_3 433.8 does not seem to belong to hydrogen, though near to it (Ångström gives $H=434.01$). A moderately concentrated solution of AuCl_3 has not shown me 433.8 with the condensed spark. In fine, the ray 433.8 is seen always, though not without difficulty, with the uncondensed spark and a very concentrated solution of AuCl_3 . No more than Demarçay, with his short coil, or Eder and Valenta have I seen this ray between poles of metallic gold, nor by the use of the condensed spark. I do not succeed in identifying it with certainty with any of the rays of Cl, H, C, N, Ar—bodies present in the spark drawn from AuCl_3 .

Potassium Sulphantimonites.—M. Pouget. — Antimony sulphite forms with potassium sulphide a normal sulphantimonite, SbS_3K_3 ; a pyrosulphantimonite, $\text{Sb}_2\text{S}_5\text{K}_4$; a metasulphantimonite, SbS_2K ; and, lastly, a compound containing still less, $\text{Sb}_4\text{S}_7\text{K}_2$.

Fluidity of Melted Nickel.—Jules Garnier.—The great fluidity of nickel when melted at high temperatures may serve to explain the increase of resistance which it gives to irons.

Combinations of Tellurium Iodide and Bromide with the Corresponding Hydracids.—R. Metzner.

Analysis of Bronzes and Brasses by the Electrolytic Process.—A. Holland. —Already inserted.

Formic Aldehyd. Action of Potassa.—M. Delépine. —Not suitable for abstraction.

Destruction of Organic Matters in Toxicology.—A. Villiers.—The author's process is founded on the use of the salts of manganese. Organs such as the liver, the spleen, and the lungs are dissolved in a few minutes. Muscular fibres are disintegrated and then dissolved in about an hour, leaving merely a fatty residue.

Caffeotannic Acid.—P. Cazeneuve and M. Haddon. —Not suitable for abstraction.

Coleopterine: a Red Pigment from the Elytra of certain Coleoptera.—Dr. A. B. Griffiths.—*Pyrochroa coccinea*, *Lina populi*, and *Coccinella septempunctata* yield one and the same pigment, which is soluble in alcohol and ether. The pigment is composed of $\text{C}_7\text{H}_8\text{NO}_5$. When isolated, the pigment is decolourised by light. Its solutions yield no characteristic absorption bands. The author names it provisionally "coleopterine."

"Fracture" of Wines.—M. Lagata.—The author refers this morbid phenomenon to the action of iron.

No. 26, June 28, 1897.

The Death of Prof. Schützenberger.—The President officially informed the Academy of the death of the late illustrious Professor. The deceased, it may be said, died in action, leaving in his laboratory important researches incomplete.

Nomination.—M. de Lapparent was elected a member of the Section of Mineralogy, *vice* the late Prof. des Cloiseaux.

Medical Use of Ozone.—Charles Chardin.—The author recommends a treatment with ozone in cases of cancer and other infectious maladies.

Researches on Nickel Steels, Permanent Magnetic Properties and Deformations.—Ch. Ed. Guillaume.

Silver Sulphantimonites.—M. Pouget.—The action of silver nitrate upon normal sulphantimonite, SbS_3K_3 , yields, according to the conditions of the operation, two well-defined bodies, $\text{SbS}_3\text{Ag}_2\text{K}$, either in an amorphous or a crystalline state. The author has only succeeded in obtaining the salt, SbS_3AgK_2 .

Part of Manganese in certain Oxidations.—Ach. Livache.—In a litharged or manganese oil, the lead or the manganese play the part of intermediaries, taking oxygen from the air and handing it over in continuous manner to the oil, which is thus oxidised more rapidly than it would be in their absence.

The Colour of the Phosphorescence of Strontium Sulphide.—José Rodríguez Mourale.—The experiments prove that the temperature at which the sulphides are formed has not a great influence on the phosphorescence, for the most phosphorescent sulphides are not those whose formation requires the strongest or the most prolonged heat. Strontium sulphide always presents a phosphorescence of a green more or less pure and intense. This property appears closely connected with the properties of production, and with substances which affect the purity of the bodies employed.

Observations on the Molecular Volumes at 0° of Various Crystallised Hydrates of Carbon.—M. Pionchon.

Trioxymethylene and Paraformaldehyd.—M. Delépine. —A thermo-chemical paper, not suitable for abstraction.

On certain Compounds of Phenylhydrazine with the Metallic Iodides.—J. Moitessier.—The compounds here discussed are the phenylhydrazinic zinc iodides, *a* and *b*; the phenylhydrazinic cadmium iodide; the phenylhydrazinic manganese iodide; and the phenylhydrazinic nickel iodide. The nitrates of the various metals of the magnesian series form crystalline compounds with phenylhydrazine.

Compounds of Metallic Salts with the Organic Bases, Homologues of Aniline, and their Isomers.—D. Tombeck.—An examination of compounds formed by toluidine, xyldine, picoline, and lutidine.

Action of Acetylene upon Silver Nitrate.—G. Arthj. —Not suitable for abstraction.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xvii.-xviii., No. 11. June 5, 1897.

Notice on the Life and Works of Alphonse Combes.—Ch. Friedel. — A long and interesting biographical sketch of the career of the late Alphonse Combes, at one time President of this Society. He was a comparatively young man, having been born in 1858, and his death was extremely sudden. The memoir is accompanied by an excellent portrait.

Reaction facilitating the Recognition of Naphthol α from Naphthol β .—E. Léger. — An aqueous solution of the matter under examination is made by triturating the crystals in a mortar. To 10 c.c. of this solution are added 2 drops of a solution of hypobromite of soda. With naphthol α a colouration, or even a dirty violet precipitate, is produced; with naphthol β a yellow colour first appears, gradually becoming greenish; it then passes to a brownish green, to again become yellow. It is of importance that all the solutions used should be freshly prepared.

On Two New Alkaloids isolated from a Species of Jaborandi.—A. Petit and M. Polonovski. — These two alkaloids have been found in a specimen of the plant for which the name *Pilocarpus spicatus* has been proposed. The alkaloid α is a colourless syrup, with a very alkaline reaction, easily soluble in water, alcohol, and chloroform. The authors have named it *pseudojaborine*; the base β , which they have named *pseudopilocarpine*, presents the same characteristics as pilocarpine, except that it does not act upon polarised light.

Contributions to the Study of Pilocarpine and Pilocarpidine.—A. Petit and M. Polonovski. — These two bodies have been the subject of a good deal of research. At the present time, however, though the properties of pilocarpine are fairly well known, those of pilocarpidine are to a certain extent under a cloud. The authors find the observations of MM. Hardy and Calmels on this subject are not sufficiently exact; this causes them to think that their theoretical conclusions are too arbitrary; they, the authors, have therefore set forth in this note the points of difference they have found, in a somewhat extended research, between their own results and those of the other afore-mentioned experimentalists.

Remarks on the Congealing-point of Milk.—J. Winter. — The author gives as a constant the temperature -0.556° , remarking that variations of 0.01° above or below this can be observed. His results have been confirmed by other workers on the subject.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Chrome Ore.—We understand that chrome ore is used to some extent in chemical works. Can any correspondent oblige with the names of any such works using this ore?—A. & Co.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1966.

SEPARATIONS WITH ALKALINE ACETATE.

By HARRY BREARLEY.

(Continued from vol. lxxv., p. 254).

II.—Nickel from Iron.

NICKEL is frequently separated from iron by repeated reprecipitation with alkaline acetate. A method previously proposed (CHEMICAL NEWS, vol. lxxiv., p. 16), in which the error introduced by using large amounts of acetate is eliminated by using large amounts of acetic acid with hydrate-free solution, worked very satisfactorily. Further experience with alkaline acetate, under more precise conditions, has shown that accuracy over a wider range may be secured without using so wastefully large a volume of reagents. The present intention is to determine, and state in definite terms, under what conditions the separation can best be made.

It would be too laborious, perhaps unnecessary, to vary the proportion of dissolved hydrate and examine each with series of free acetic acid and acetate. The successful separation in solutions containing no dissolved hydrate which had been formerly experienced led me to work out the series tabulated below:—

TABLE III.

Acetic acid.	Ammonium acetate.					C.c.	Range of temperature turbidity. °C.
C.c.	140.	160.	180.	220.	260.	400.	
0	33'0	31'66	30'55	—	—	—	86—68
10	—	32'82	31'96	30'4	—	—	87—76
20	—	—	32'7	31'77	31'1	—	90—82
40	—	—	—	32'47	32'0	30'6	29'89 91—78
60	—	—	—	—	32'68	31'57 30'96	95—83

The work was done on a 5 per cent nickel steel. The figures represent c.c.'s of the potassium cyanide used in titrating the filtrate.

A shorter series was gone through in which the acetic was replaced by hydrochloric or nitric acid, and another in which the sample was dissolved in nitric acid instead of aqua regia. The results obtained were quite as good as those tabulated, but no marked improvement on them. The separations with soda acetate were slightly better than those with ammonium acetate.

Separations from solutions containing the maximum amounts of dissolved hydrate gave—

TABLE IV.

Acetic acid.	Acetate.			C.c.
	5.	10.	20.	
0	0'0498	0'0483	0'0477	
10 {	—	0'0500	0'0498	
	—	0'0504	0'0504*	

The line marked with an asterisk refers to soda salts. The samples actually contained 0'0500 grm. Ni. Experiments were made with solutions containing half the dissolved hydrate. They were neither better nor worse than those already tabulated.

It became necessary to choose what amounts of dissolved hydrate, acid, and acetate should be adopted in order that the effect of certain other variations might be observed. I believe that almost any volume of acetate, with a suitable amount of free acid, could be satisfactorily employed; and, similarly, any proportion of dissolved hydrate might be fixed upon. Chiefly in deference to

customary practice I chose solutions containing the total soluble hydrate.* But such solutions have an irritating tendency to become turbid if they are left for any length of time at this stage†; and, moreover, a few c.c. of such dilute acetate as is used made a deal of difference to the separation. For these reasons I add also 10 c.c. of acetic acid. The choice proved a good one.

I will now describe the method for separating nickel and iron. The details rest upon observations either already made or subsequently to be made. It is written as applying to alloys of the two metals; it can, of course, readily be adapted to those elements in other states.

Dissolve in hydrochloric acid and oxidise with nitric acid. In case the alloy contains little or no carbon it may be straightway dissolved in nitric acid. Dilute, cool, add carbonated alkali until a slight permanent precipitate forms (if the sample is always dissolved in the same amount of acid the volume of standard alkali will be approximately known); add 10 c.c. acetic acid, dilute to somewhat less than a litre (say) with either hot or cold water, add 10 to 12 c.c. ammonia (soda) acetate, of the strength previously mentioned, for each grm. of iron in solution. If, on nearing the boiling-point, owing to imperfect neutralisation, no turbidity occurs, add acetate (2 or 3 c.c. at a time) until it does. Boil, measure volume, cheese until precipitate settles, and filter aliquot part through asbestos. Then cool, neutralise, add measured quantity of dilute ammonia, titrate with KCN and AgI, or estimate Ni in any other way.

The following variations show that the method is widely applicable. They are a selection only from a list embracing every likely variation I could think of.

Dr. Wolcott Gibbs (CHEMICAL NEWS, vol. xi., p. 102; Crookes, "Select Methods," 3rd edition, p. 227), respecting the separation of iron and nickel says:—"The solution from which the iron is to be precipitated must be dilute. Half a litre should not contain more than one grain (0'065 grm.) of the sesquioxide." From a solution containing $\frac{1}{2}$ grm. of iron ($\frac{7}{8}$ grains) to the half litre, I have already separated nickel up to 5 per cent of the alloy. As the separations of nickel from iron by means of soda phosphate in acetic solutions is said to be applicable only when the nickel does not exceed 3 per cent ("Select Methods," p. 205), it was anticipated that with higher percentages the separation with acetate would become less accurate. This fear was not realised, at least not with the somewhat abnormal quantities shown below. Each sample contained 1 grm. of iron.

TABLE V.

Ni taken.	Ni found.	Per cent recovery.
0'1000 grm.*	0'1002	100'2
0'2000	0'2008	100'4
0'3000	0'3009	100'3
0'5000	0'4993	99'86
0'8000*	0'8008	100'1

These were done with ammonia salts. Soda salts were tried in two cases only, and gave—Nickel taken, 0'1000, 0'2000 grm.; Ni found, 0'1002, 0'1996. The potassium cyanide was standardised with quantities of nickel corresponding to that in the filtrates marked with an asterisk. The values for the KCN obtained were not quite proportional. The variation was nearly 1 per cent.

* The "Foreign Science" letter in the CHEMICAL NEWS, vol. xvii., p. 286, chronicles experiments which are interesting in relation to the remarks on dissolved hydrate in my previous paper. It is related that M. Jeannel prepared in the cold a solution of ferric chloride containing nine times as much iron as the official solution. "A few drops thrown into water produce a voluminous brown precipitate. The solution of ferric chloride is decomposed and precipitated by small quantities of sulphuric acid or of sulphates. It is likewise decomposed by citric, tartaric, or by a few drops of HCl or HNO₃."

† Boiling at this point without the addition of any further reagent forms what is known as Herschel's or Schwarzenberg's method. Dittmar ("Quant. Chem. Anal.," p. 74), in 1887, speaks of it as "the best method." (See also "Fresenius' Quant. Anal.")

The values for the intermediate tests were calculated by interpolation from those found experimentally. No proportions of the two metals less favourable to a perfect separation than the above are likely to be found in practice.

If one has not long been used to perfectly neutralising iron solutions, it will frequently happen, through imperfect neutralisation, that the acetate prescribed is insufficient to even cause a turbidity at boiling-point. In such cases could more acetate be added without introducing error? It has been repeatedly pointed out that the acetate should be added to the cold solution. This precaution probably arose through the almost universal custom of adding wastefully large amounts of acetate. The following experiments answer the question, at least for the present elements and mode of operating.

Two samples containing 1 grm. iron and 0.1 grm. Ni, and in other ways treated as usual, except that no acetate was added, were heated to 90–100° C.; 13 c.c. and 15 c.c. were dropped from a burette, the solution being meanwhile vigorously stirred. Each drop as it touched the iron solution formed a precipitate which was immediately dissolved in the agitated liquid. In the first case a faint turbidity, and in the second case a decided turbidity, was apparent before the last drop of acetate was delivered. There was recovered 0.1001 gr. nickel in each case. The dropping of the acetate and stirring of the liquid seem needless precautions. Two samples, prepared as before, were heated to 97° C., and 15 c.c. acetate were run in and allowed to become mixed by the convective movement of the liquid. The amounts of nickel recovered were 0.1003 and 0.1001 grm. respectively.

When too much acetate has been inadvertently added, and the iron is precipitated at low temperatures, more acid may be added, the assay re-started; or, where neither is desirable, the heating should be discontinued as soon as the precipitate gathers into flocks and the solution against a white rod shows itself only slightly coloured; the flask well shaken, cheesed, &c., as usual. In this way the recovery is greater than if the solution is raised to boiling, although it is not necessarily a complete recovery. On the other hand, if the solution is turbid at boiling-point, but the precipitation of the iron not so complete as to give a clear filtrate, instead of adding more acetate the iron may be further precipitated by prolonged boiling. The table shows that no error is thereby introduced. One grm. iron present in each case.

TABLE VI.

Minutes boiled.	Ni present.	Ni found.
1	0.1000	0.1003
40	0.1000	0.1001
50	0.1000	0.1003

Even when there is no precipitate at boiling-point the separation may be effected by partially confining the steam, and thus raising the temperature. But such an arrangement is troublesome, and has been of no service yet. Under other circumstances it may be.

Additional quantities of ammonium nitrate or chloride act in two ways. They lessen the quantity of alkali necessary to neutralise the iron solution, and they lower the temperature of turbidity. In the accompanying table there was added to I. nothing; to II. ammonium chloride equal to 10 c.c. HCl; and to III. ditto equal to 20 c.c. HCl. The same vol. of acetate, 14 c.c., was added in each case.

TABLE VII.

	Ni added.	Ni found.
I.	0.1000	0.0998
II.	0.1000	0.0998
III.	0.1000	0.0996

Jewett (CHEMICAL NEWS, xl., 273) finds that if ammonium chloride is present the separation of Ni and Fe is more complete than otherwise.

If the nickel is to be estimated by titration with cyanide and silver iodide, it is but a slight disadvantage should the filtrate, owing to imperfect separation of iron, be slightly tinted. Such small amounts may be separated by adding ammonia, or more acetate and boiling and filtering, or by proceeding with the titration as usual. In the latter case, where so much is present as to interfere with the end reaction, a slight excess of the KCN may be added, the solution run through an asbestos filter, and the clear filtrate taken to faint turbidity with the silver nitrate. Comparatively large quantities of suspended ferric hydrate are known to introduce no inaccuracy in cyanide titrations of cupric ammonium solutions, so that examples to justify the practice with nickel are unnecessary.

Obvious enough is the suggestion that in precipitating the iron with alkali and acetate, or alkali alone, adding excess KCN and going back in a filtered aliquot part with silver nitrate, lies a rapid approximate method, such as might be serviceable where a large number of assays had to be completed in little time. Precipitating with ammonium carbonate, adding ammonia, and titrating as just suggested, yielded 99 per cent of the Ni present. The separation was better in cold than in hot solutions, and better when the precipitation was made with carbonate than with ammonia.

There are elements sometimes associated with nickel in the filtrate which are not without action on the cyanide. Of zinc and cobalt, which rarely appear in steel works' laboratories, nothing need be said now. Such quantities of manganese as are found in steel have no influence whatever when ammonia salts are used throughout. But the titrating of a filtrate containing severally 0.05 grm. of nickel and manganese begins to present difficulties when separated from iron as previously described. The ammonia salts present are unable to keep the manganese in solution for any considerable time. In presence of larger quantities of ammonia salts a satisfactory titration may be made with double this quantity of manganese present. I need scarcely point out that if soda salts are used the manganese must be separated at some stage prior to the titration, as suspended oxide of manganese causes low results (CHEMICAL NEWS, i., 25; xxxiii., 152). Copper is conveniently separated from the filtrate as sub-sulphocyanide ("Select Methods," p. 294). Chromium, in the presence of large amounts of acetic acid, would be partly in the filtrate, but with the amount used (10 c.c.) appears to be altogether precipitated with the iron. Large amounts of aluminium would demand exceptional treatment. I propose to postpone this item until the separation of aluminium and iron is considered.

ADDENDA.

Remarks on the Titration.

The means adopted in this paper of estimating the nickel have been largely used and highly spoken of during the past few years. Titration with KCN, in which the indicator was the solubility of the previously formed precipitate in an excess of the reagent, or the decolouration of added cupric ferrocyanide, had been previously used. The use of silver iodide, formed in the solution, as indicator makes the titration as perfect as can be. Denigès (Comptes Rendus, cxvii., p. 1078; CHEMICAL NEWS, lxix., p. 42) shows how any compound of silver, or anything capable of modifying such a compound, can be accurately estimated by these means. Moore (CHEMICAL NEWS, lxxii., p. 92) gives a good account of nickel titration after separating that element from steel. The method has also been adopted in the same connection by American writers (Campbell and Andrews, Journ. Amer. Chem. Soc., Feb., 1895).

Before entering on the work previously described, the titration was performed in the presence of varying amounts of reagents. They are not altogether without influence. The presence of ammonium salts deepens the turbidity formed by the silver nitrate and potassium iodide. If

equal volumes of sulphuric, nitric, and hydrochloric acid are added to separate solutions before neutralising, the iodide precipitate is decreasingly turbid in the same order. Hence, as has been previously recommended, about 2 c.c. of H_2SO_4 should be added unless large quantities of ammonium (or soda) salts are already present.

It is well known that very large quantities of ammonia introduce error into the titration; however, statements on this head are not very precise, and leave much to individual discretion. Denigès says "Variations even very considerable in the proportions of ammonia added, the presence of potash or soda, free or as salts, have no influence on the quantity of silver nitrate used." Campbell and Andrews — "The solution should have just enough free ammonia to give a slight but distinct odour"; and so on. To test the point, ammonia (0.880) was diluted with three times its volume of water. A solution was prepared containing ammonium sulphate, potassium iodide, and ammonia to faint alkalinity, then 4 c.c. in addition, and made up to two litres. 100 c.c. of this mixture was made up to 300 c.c. with water, a measured amount of silver nitrate, and varying amounts of ammonia. The table shows the result with the indicator alone, and ditto to which has been added 0.01 grm. nickel. The two sets of solutions were not identically alkaline to begin with.

Ammon. added	0	$\frac{1}{2}$	2	5	10	20	50 c.c.
C.c. Indicator	5.38	5.03	4.93	4.88	4.88	4.68	4.43
KCN Do. + Ni	19.27	19.0	19.0	18.95	18.94	18.59	10.6(1)*

* This was several times repeated.

The chloride, nitrate, and acetate of ammonia are without any appreciable influence.

A few drops of artificial alizarin solution may be used as indicator when neutralising the free acid. In an alkaline solution it has a pink colour, and this, along with the creamy silver iodide, produced a purple tint, which changes to pink again when the AgI is dissolved. It is easy to titrate by gas-light in this way.

Moore finds that when the temperature of the solution exceeds 20°C ., the titrations are irregular. Some laboratories are frequently hotter than this. Solutions which have stood about in our own are at present 25°C ., but no irregularity has been noticed on this account. Titration certainly ought not to be performed in hot solutions, because else the KCN is decomposed apart from any metal which may be in solution, nor should solutions be unnecessarily exposed to the air, because they absorb CO_2 and give off HCN; hence the slight perceptible odour (Gmelin, vii., 476). But Denigès finds that, in presence of fixed alkali,—"soap-boilers' lye,"—a solution of KCN can be kept unimpaired for an indefinite time, and even boiled for five minutes without appreciable alteration.

I am obliged to Mr. H. Jarvis for material, though indirect, assistance.

ON THE

VOLUMETRIC DETERMINATION OF ZINC BY POTASSIUM FERROCYANIDE.

By L. L. DE KONINCK and EUG. PROST.

(Concluded from p. 39).

Application of the Method to the Estimation of Zinc in Minerals, &c.

THE titrimetric estimation of zinc by ferrocyanide in acid solution, in minerals and zinciferous metallurgical products, necessitates the preliminary removal of metals susceptible of reacting with the ferrocyanide. The method is approximately the same as that adopted for preparing the solution when using Schaffner's method. To the ammoniacal solution finally obtained, containing a more or less constant quantity of ammoniac compounds, are

added a few drops of sodic sulphite; it is then neutralised with hydrochloric acid; then acidulated with a quantity of the same acid, which quantity should be kept constant.

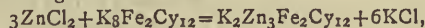
To this solution is added a measured volume of ferrocyanide, constituting an excess of 20 to 25 per cent on the quantity necessary for exact precipitation. After digesting for ten or fifteen minutes, the back titration is performed by means of a neutral or slightly acid titrated solution of ZnCl_2 . The quantity of zinc corresponds to the excess of the reagent.

Solutions employed.

A. A zinc solution $\text{TZn} = 0.01$ grm.—that is to say, 10 grms. per litre; it should be slightly acid. This solution is made by dissolving 10 grms. of pure zinc in the smallest possible quantity of hydrochloric acid, in a litre flask, at a moderate temperature. When the solution is complete we bring the volume up to about half the final, neutralise the excess of acid with potassic carbonate, until there is a faint permanent precipitate, which disappears on adding a drop or two of hydrochloric acid. The solution is then cooled to the normal temperature, and the flask filled exactly to the mark with distilled water.

B. A solution of ferrocyanide of potassium. Although in all our experiments we used normal solutions, we think it would be an advantage to give this solution the titration $\text{TZn} = 0.00625$ grm., or $\frac{1}{2}$ more than the titration 0.005. When, as is frequently the case in industrial assays, the quantity of zinc present is approximately known, it is best to use 2 c.c. of ferrocyanide per centigram. of zinc expected, and we should then have the excess of about 25 per cent that we recommend.

According to the formula—



the ferrocyanide solution ought to contain per litre the weight given by the proportion—

$$3\text{Zn} : \text{K}_3\text{Fe}_2\text{Cy}_{12}, 6\text{H}_2\text{O} = 625 \text{ grms.} : \text{X} = 26.99 \text{ grms.}$$

$$195.33 : 843.52$$

This is therefore the weight to take if we have perfectly pure ferrocyanide; if not, we take a little more, and after having found the exact titration of the liquid we dilute it to bring it to the titration wished for. Let us further remark that, on account of the influence of the acid, and of the ammonium chloride, which is generally present in the solutions under examination, it is preferable in every case to determine the titration by direct experiment, and also that it is by no means necessary to know exactly the titration of the liquid, if we do the assays by comparison with pure zinc. In such a case it would suffice to prepare the reagent by dissolving 27 grms. of commercial ferrocyanide weighed on the assay balance.

C. The indicator. A 1 per cent aqueous solution of crystallised nitrate of uranium.

Experimental Determination of the Titration of the Solution of Ferrocyanide, and its Volumetric Relation to the Zinc Solution.

With a view to counterbalancing the influence of acid and salts of ammonia on the estimations, it is important that the determination of this relationship should be made under conditions as similar as possible to those of an assay; we therefore proceed as follows:—

Twenty c.c. of ZnCl_2 , 100 c.c. of water, 50 c.c. of AmCl ,* 2 drops of sulphite,† and 10 c.c. of hydrochloric acid ‡ are poured into a flask.

* A solution of AmCl , containing 200 grms. per litre = 3.75 normal. The proportion of AmCl is considerable; it would certainly be preferable to employ less, if possible, but we are obliged to use this quantity in order to reach the proportion which accumulates in an assay by the neutralisation of the ammoniacal solution.

† A solution of 10 grms. of $\text{Na}_2\text{SO}_3, 7\text{H}_2\text{O}$ per 100 c.c. of water. As very little of this is used, and as it changes in contact with the air, not much should be prepared at a time.

‡ Pure HCl , sp. gr. 1.075. This acid is approximately 5 normal and contains about 182 grms. of HCl per litre.

With the exception of the ZnCl_2 solution, which should be measured with an accurate pipette, the measurements may be made with an ordinary graduated glass.

Into this mixture we pour 40 c.c. of ferrocyanide, constituting an excess of 25 per cent; stir well, and let stand ten or fifteen minutes. The precipitate, which was at first gelatinous, has by this time become coherent and settled. We then titrate back with the zincic solution, stirring well after each addition; a tube is more convenient than a rod for the touch test.

After each addition of zincic chloride, a drop of the mixture is taken and let fall into a drop of the indicator. We regulate the quantity of ZnCl_2 to be added each time by the intensity of the colour produced by contact with the uranium salt, and stop when the addition of one or two drops no longer gives a brown colouration, even after two minutes contact with the indicator; this point is shown with great clearness.

The operation we have just described enables us to establish once for all the relation of the value of the two solutions and the titration of the ferrocyanide; in fact, this solution will keep without any change if we keep it away from the light. The slight amount of ferrocyanide which may be formed is without influence on the assay if—as we have shown—we add a small quantity of sodic sulphite to the solution to be titrated, as the ferri- is then re-converted into ferrocyanide.

Assay of Minerals.

Preparation of the Solution.—We treat a sample of 2·5 grms. of the mineral, dried at 100° , with nitric acid, in the case of a blende; or with fuming hydrochloric acid, if we have to do with a calamine. After complete solution we evaporate to dryness, so as to make the silica insoluble, and take up the residue with 5 c.c. of fuming hydrochloric acid and a little water; then, after well heating so as to dissolve the basic salts, add 50 or 60 c.c. of water and heat to 70° . We then pass a current of sulphuretted hydrogen; during the passage of the gas we gradually add 100 c.c. of water, to facilitate the precipitation of the lead and cadmium, which would not come down in a too acid solution; but we should not, on the other hand, keep up the current of gas beyond the necessary time, nor dilute the solution too much, or some of the zinc might be precipitated. The precipitated sulphides are collected on a filter, with the silica, if we do not want to estimate the latter separately; and washed with 100 c.c. of water, to which is added 5 c.c. of hydrochloric acid charged with sulphuretted hydrogen.

The washing is complete when the last drops, made alkaline with ammonia, no longer give the slightest precipitate with sodic sulphide; the filtrate is then boiled until all the sulphuretted hydrogen is driven off. We then add 10 c.c. of fuming hydrochloric acid, and 10 to 25 c.c. of saturated bromine water, according to the quantity of iron present, so as to re-oxidise the ferrous salts and to assist the precipitation of the manganese; it is then poured, a little at a time, with constant shaking, into a 500 c.c. matras, containing 100 c.c. of strong ammonia* and 10 c.c. of a 20 to 25 per cent of bicarbonate of ammonia.

On cooling we add water up to the mark, shake well, let the precipitate settle, and filter, but taking care that the filter and all the apparatus is dry.

This method is one which would be used for the most complex minerals. The addition of bromine would not be necessary if there were no manganese present.

We take 100 c.c. of the ammoniacal filtrate, add a few

drops of sulphite, add hydrochloric acid (sp. gr. 1·075) until neutral (about 30 c.c.), then add 10 c.c. more of the same acid.

If after acidulating the liquid becomes brown, owing to the liberation of bromine, it is because the quantity of sulphite is insufficient, and it is then necessary to add a little more than sufficient to cause the disappearance of this brown colour.

Into the solution thus prepared we run such a volume of ferrocyanide, that there may be an excess of 20 to 25 per cent on the quantity necessary for the exact precipitation of the zinc contained in the 100 c.c. of the ammoniacal solution used for the test.

These 100 c.c. contain the zinc from 0·5 grm. of mineral. It is easy to understand, after what we have said previously *à propos* of the value of the titration chosen for the ferrocyanide, that it is necessary to use here a volume represented by the centesimal contents; for example, 38 c.c., if we are dealing with a mineral containing 38 per cent. In practice, we take it as 40 c.c. in round numbers. This has the advantage of permitting the use of calibrated pipettes, which are more easily used than a burette for repeated measurements of the same solution, and also of giving, we think, more accurate results when working quickly.

If we do not know sufficiently nearly the value of the ore, we do a direct assay to determine the volume of the ferrocyanide to be used. For this purpose we take 50 or 100 c.c. of the ammoniacal solution, treat as above, then run in ferrocyanide a little at a time, shaking well, until a test with nitrate of uranium gives, even when tried after waiting some instants, a distinct brown colouration, indicating the presence of a faint, but appreciable, excess of the reagent.

We should use for the final assay a volume higher by one-fifth to one-fourth than that used for the preliminary test; or double this, of course, if we were working on 50 c.c. only. The assay is finished after digesting ten or fifteen minutes, by titrating back to find the excess of ferrocyanide.

By subtracting the weight of the zinc contained in the quantity of the zincic chloride used in titrating back (10 m.grms. per c.c.) from the weight corresponding to the ferrocyanide, we obtain that contained in 0·5 grm. of the mineral; this latter multiplied by 2 gives the centesimal contents in centigrams.

In assays requiring the highest degree of exactitude, such as in cases of dispute, it is preferable to work by comparison; that is to say, to make two parallel estimations—one on the mineral itself, the other on an artificial solution imitating as nearly as possible the value in zinc of the mineral. In other words, on a solution of pure zinc containing as nearly as possible a weight of zinc equal to the weight of zinc in the sample taken of the ore.

This artificial solution is prepared by dissolving in a 500 c.c. flask as much zinc as can be taken up by 20 c.c. of fuming hydrochloric acid, adding to this 100 c.c. of water, then 100 c.c. of strong ammonia and 10 c.c. of the solution of carbonate of ammonium, as with the mineral; after cooling, make the volume up to 500 c.c. with distilled water.

We know that the hydrated precipitates of iron and aluminium produced by ammonia in metallic solutions retain a quantity of zinc by no means negligible (E. Prost and Hassreider, *Zeit. Anorg. Chem.*, 1892). To obtain complete separation it is necessary to have recourse to double precipitation, which complicates the operation.

By working by comparison we can avoid this complication by adding to the artificial solution, before treating with ammonia, such a volume of ferric chloride of known strength that the weight of iron thus introduced shall be as nearly as possible equal to that contained in the sample of ore taken.*

* Sp. gr. 0·93, or approximately 10 normal. It is desirable to use the smallest possible quantity of both ammonia and of bicarbonate, so as to keep down the contents of ammonium chloride in the final liquid; but it would be well to know if this diminution might not augment the quantity of zinc carried down by the hydrated precipitates of iron and aluminium. Not having had the time to thoroughly study this question, we have adhered to the method generally employed at the present day, for the preparation of the solution for an estimation by Schaffner's method.

* To facilitate calculation, it is best to prepare a solution containing 10 or 25 grms. of iron per litre, and take the necessary volume by means of a graduated pipette.

In this manner the influence of the iron will be neutralised. We operate side by side on the two solutions, taking 100 c.c. of each; acidulating them both with the same quantity of hydrochloric acid, adding the same volume of ferrocyanide, and titrating back with the same solution of zinc chloride after sufficiently digesting the precipitate.

The centesimal value, expressed in centigrams., will be found by the expression—

$$\text{Zn per cent} = 2[0.2 P + 0.01 (N - N')];$$

in which P represents the weight of zinc used for the preparation of the artificial solution, N and N' the number of c.c.'s of the zinc solution at 0.01 of zinc per c.c. used for the back titrations respectively of the 100 c.c. of artificial solution and the 100 c.c. of the solution of the mineral. In fact, as in both cases we have used the same volume of ferrocyanide, the weights of zinc contained finally in each experiment should be equal. For the artificial solution this weight is—

$$\frac{1}{2} P = 0.2 P,$$

augmented by 0.01 grm. for every c.c. of chloride of zinc used; if, for example, it be 0.01 N, then the total will be $0.2 P + 0.01 N$.

For the mineral the weight of zinc will be that contained in 0.5 grm. of the sample; we represent it by X, also augmented by 0.01 grm. for each c.c. of zinc chloride used; this will be $X + 0.01 N'$. From this we get the equation—

$$\begin{aligned} X + 0.01 N' &= 0.2 P + 0.01 N \\ X &= 0.2 P + 0.01 (N - N'). \end{aligned}$$

When working with 50 grms. this must be doubled. It must not be overlooked that if N' be greater than N, the expression $0.01 (N - N')$ becomes negative, and the corresponding quantity must be subtracted from $0.2 P$.

Examples.

I. If the ore contains about 40 per cent zinc, to prepare the artificial solution we take—

$$P = 1.0 \text{ grm.} = (2.5 \times 0.40);$$

then if N = 4.7 c.c. and N' = 4.1 c.c., the zinc present will be—

$$2[0.2 \times 1.0 + 0.01(4.7 - 4.1)] = 2[0.2 + 0.006] = 0.412, \text{ or } 41.2 \text{ per cent.}$$

II. If the ore contains about 30 per cent of zinc, for the artificial solution we take $P = 0.75$ grm. Then let N = 2.3 c.c. and N' = 3.9 c.c., the zinc present will be—

$$2[0.2 \times 0.75 + 0.01(2.3 - 3.9)] = 2[0.15 - 0.016] = 0.268, \text{ or } 26.8 \text{ per cent.}$$

Direct Titration.

For ordinary daily assays, not requiring such extreme accuracy, we can use direct titration. For this purpose we run the carefully titrated ferrocyanide into the acid zinc solution (warmed to 60° or 70°) until a touch test with nitrate of uranium gives a faint brownish colour. We do not consider the reaction at an end until two touch tests, done at an interval of two or three minutes, both give a colour, and, further, that after the addition of two or three drops of ferrocyanide the touch test should give a distinctly stronger reaction. In direct titration we prefer to use a solution of ferrocyanide, $\text{TZn} = 0.01$ grm., corresponding to the solution of zinc chloride.

Finally, we give the results obtained by us, by V. Hassreidter, and by Van de Castele, using our process on several ores. In our own assays we did the back titration with a $\frac{1}{2}$ normal solution of chloride of zinc, $T = 0.01628$ grm.

The ferrocyanide solutions were various and were used in varying quantities; but as all the assays were done by comparison with an artificial solution, and as in every analysis the volume of the two solutions was the same,

it is unnecessary to give the strength and volume of this reagent.

The values given immediately after the description of the mineral are those found by Schaffner's process in an independent estimation by ferrocyanide.

I.

Roasted ore, free from copper, &c.	..	..	45.28, 45.3% Zn.
Artificial solution	..	..	1.125 grms. of pure Zn.
ZnCl ₂ used in titrating back	..	..	2.80, 2.92, 2.84, 2.89 c.c.
Mean	..	..	2.86 c.c.
Ore used	..	..	2.5 grms.
ZnCl ₂ used in titrating back	..	..	2.73, 2.70, 2.70
Zn per cent..	..	..	45.42, 45.52, 45.52

II.

Roasted plumbiferous blende	..	..	62.86% Zn.
Artificial solution	..	..	1.5 grm. of pure Zn.
ZnCl ₂ used in titrating back	..	..	6.02, 6.08, 6.05, 6.04 c.c.
Mean	..	..	6.05 c.c.
Ore used	..	..	2.5 grms.
ZnCl ₂ used in titrating back	..	..	5.18, 5.15, 5.08, 5.16
Zn per cent..	..	..	62.83, 62.93, 63.16, 62.89

III.

Cupriferous calamine..	..	..	24.30% Zn.
Artificial solution	..	..	0.6 grm. pure Zn.
ZnCl ₂ used in titrating back	..	..	11.90, 11.95 c.c.
Mean	..	..	11.925 c.c.
Ore used	..	..	2.5 grms.
ZnCl ₂ used in titrating back	..	..	11.90, 11.87, 11.92, 11.95
Zn per cent ..	..	..	24.10, 24.20, 24.04, 23.94

IV.

Crude calamine	..	..	55.80% Zn.
Artificial solution	..	..	1.375 grm. of pure Zn.
ZnCl ₂ used in titrating back	..	..	2.29 c.c.
Ore used	..	..	2.5 grms.
ZnCl ₂ used in titrating back	..	..	2.20, 2.18
Zn per cent ..	..	..	55.30, 55.35

V.

Calamine No. 1 (Hassreidter) ..	..	48.3 per cent Zn.
By the ferrocyanide process	..	48.1 " "

VI.

Calamine No. 2 (Hassreidter) ..	..	51.65 " "
By the ferrocyanide process	..	51.59 " "

VII.

Blende No. 1 (Hassreidter) ..	..	36.50 " "
By the ferrocyanide process	..	36.80 " "

VIII.

Blende No. 2 (Hassreidter) ..	..	42.45 " "
By the ferrocyanide process	..	42.15 " "

IX.

Van de Castele ..	..	38.29 " "
By the ferrocyanide process (1st assay)	..	38.34 " "
	..	38.27 " "
" " (2nd assay)	..	38.41 " "
	..	38.39 " "

The concordance between the different assays of the same mineral shows beyond a doubt that Galletti's method, as we have modified it, merits as much confidence as that of Schaffner. The almost absolute concordance of the results we obtained in the five assays relative to the influence of nitrous acid—minimum, 33.88 c.c.; maximum, 33.94 c.c. ZnCl₂—the last done before we turned to minerals, show the exactitude of which our method is susceptible when used in experienced hands, while the results obtained by MM. Hassreidter and Van de Castele prove that long experience is not necessary in order to obtain satisfactory results. We consider our process to be much simpler and quite as quick as that of Schaffner. We leave it to our confrères, whose position

necessitates them making frequent estimations of zinc, to decide if one of these processes should replace the other, trusting that they will give them an impartial trial.

ACTION OF PHOSPHORUS PENTACHLORIDE ON ANILINE AND ITS SALTS.*

By J. ELLIOTT GILPIN.

(Concluded from p. 44).

Analysis of the Dry Powder.

Preparation I.

The specimen of aniline used for this was not purified and was very dark, and the products retained some of the impurities.

0.2332 grm. of the substance gave 0.0746 grm. AgCl.
0.19765 grm. of the substance gave 0.0631 grm. AgCl.
0.2102 grm. of the substance gave 0.0553 grm. $Mg_2P_2O_7$.
0.154 grm. of the substance gave 0.3724 grm. CO_2 .
0.1974 grm. of the substance gave 0.4784 grm. CO_2 .

Preparation II.

0.2366 grm. of the substance gave 0.0784 grm. AgCl.
0.2366 grm. of the substance gave 0.0595 grm. $Mg_2P_2O_7$.
0.2096 grm. of the substance gave 0.0691 grm. AgCl.
0.2096 grm. of the substance gave 0.0552 grm. $Mg_2P_2O_7$.
0.488 grm. of the substance gave 0.0633144 grm. N (Kjeldahl).
0.189 grm. of the substance gave 22.23 c.c. N at 25° and 758.8 m.m.
0.1584 grm. of the substance gave 0.3863 grm. CO_2 .

Preparation III.

0.1966 grm. of the substance gave 0.0655 grm. AgCl.
0.2025 grm. of the substance gave 0.494 grm. CO_2 .

Analyses of the Crystals.

0.2258 grm. of the substance gave 0.057 grm. $Mg_2P_2O_7$.
0.2309 grm. of the substance gave 0.0792 grm. AgCl.
0.2309 grm. of the substance gave 0.0612 grm. $Mg_2P_2O_7$.
0.2324 grm. of the substance gave 0.0792 grm. AgCl.
0.1613 grm. of the substance gave 0.0553 grm. AgCl.
0.1613 grm. of the substance gave 0.0435 grm. $Mg_2P_2O_7$.
0.1483 grm. of the substance gave 0.0503 grm. AgCl.

	Calculated for $PCl(NHC_6H_5)_4$.	Found (powder).					
		Preparation I.		II.		III.	
Cl	8.14	7.90	7.89	—	8.19	8.14	8.24
P	7.13	—	—	7.44	7.02	7.36	—
N	12.89	—	—	—	12.97	13.04	—

	Calculated for $PCl(NHC_6H_5)_4$.	Found (crystals).					
		Preparation I.		II.		III.	
Cl	8.14	—	8.47	8.42	8.47	8.38	—
P	7.13	7.05	7.40	—	7.53	—	—
N	12.89	—	—	—	—	—	—
C	66.29	65.94	66.09	—	66.50	66.52	—

Attempts made to substitute a residue of aniline for the chlorine atom in the compound, $PCl(NHC_6H_5)_4$, have up to the present been unsuccessful. Some of the powder was added to boiling aniline, in which it readily dissolved; but the product which separated out when it cooled had the same composition as the original material.

Action of Sulphuric Acid on Chlorphostetranilide, $PCl(NHC_6H_5)_4$.

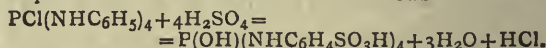
When chlorphostetranilide is treated with concentrated sulphuric acid, hydrochloric acid is given off, and the substance goes into solution. If the acid is warmed and saturated with the tetranilide it becomes solid, with crys-

tals, on cooling. The following method was used for the preparation of the compound formed in the action:—

The tetranilide was treated with as small a quantity of sulphuric acid as possible, and the solution warmed to complete the decomposition and expel the hydrochloric acid. After cooling, water was added, in volume about twice that of the acid, and the solution heated to dissolve the substance precipitated by the water. This solution, on standing, deposits clear octahedral crystals, which can be filtered off, and re-crystallised from water. These crystals are extremely efflorescent, and begin to lose their water of crystallisation before the water which they hold mechanically can be removed.

It has acid properties and is very soluble in water and alcohol, as are also the barium and lead salts, which are formed by neutralising a solution of the acid with barium and lead carbonates, and filtering and evaporating the solutions.

On account of the ease with which these salts dissolve in water, they could not be obtained in a crystallised condition suitable for analysis, but in each case were evaporated to dryness over the water-bath. The action of the sulphuric acid on the tetranilide is as follows:—



The sulphur and phosphorus were determined as barium sulphate and magnesium pyrophosphate respectively. The substance was decomposed according to the Pierson method with nitric acid and potassium chlorate.

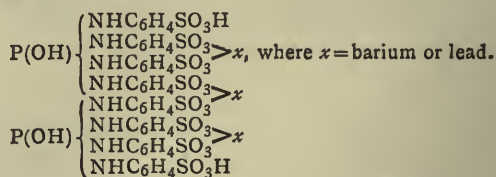
0.1518 grm. of the substance gave 0.1927 grm. $BaSO_4$.
0.2086 grm. of the substance gave 0.0313 grm. $Mg_2P_2O_7$.

		Calculated for $P(OH)(NHC_6H_5SO_3H)_4$.	Found.
S		17.39	17.44
P		4.21	4.19

The determinations of the barium and lead in the salts gave the following results:—

0.4358 grm. of the substance gave 0.1614 grm. $BaSO_4$.
0.4499 grm. of the substance gave 0.1694 grm. $BaSO_4$.
0.6464 grm. of the substance gave 0.2741 grm. $PbSO_4$.
0.6333 grm. of the substance gave 0.2676 grm. $PbSO_4$.

These results cannot be explained on the assumption of the formation of a simple compound; but they agree with a salt of the following composition, in which part of the hydrogen of two molecules of the sulphonic acid is replaced by the metals:—



	Calculated for these salts.	Found.	
		1.	2.
Ba		21.89	21.78
Pb		29.69	28.95

Action of Phosphorus Pentachloride on the Toluidines.

When the three toluidines are treated under the same conditions as the aniline with phosphorus pentachloride, they give compounds analogous to chlorphostetranilide. The method of purification was the same as that used with the aniline compound. The results of the analyses were as follows:—

Orthotoluidine.

0.2011 grm. of the substance gave 0.0599 grm. AgCl.
0.2888 grm. of the substance gave 0.0692 grm. $Mg_2P_2O_7$.

Metatoluidine.

0.213 grm. of the substance gave 0.066 grm. AgCl.

Paratoluidine.

0.2918 grm. of the substance gave 0.0814 grm. AgCl.

	Calculated.	Found.		
		Ortho.	Meta.	Para.
Cl ..	7.21	7.36	7.65	6.89
P ..	6.32	6.29	—	—

Besides the compounds already mentioned, two other substances have been obtained by treating aniline with phosphorus pentachloride, under different conditions. The results of the investigation carried out on these substances will be published shortly.

ALUMINUM ALCOHOLATES.

By H. W. HILLYER.

In a former paper (*Amer. Chem. Journ.*, xix., p. 37) we detailed the results of a research in regard to the action of aluminum on ethyl alcohol to which a small amount of fuming stannic chloride had been added. Since sending that paper to the publisher, other observations have been made, which add somewhat to our knowledge of this reaction; and similar studies have been made with methyl alcohol and the two propyl alcohols.

As stated in the article referred to, various anhydrous chlorides, which are soluble in alcohol, were used in an attempt to avoid a difficulty encountered in using mercuric chloride. When absolute alcohol is poured upon chipped aluminum, and either platinic chloride, mercuric chloride, or stannic chloride is then added, a rapid deposition of the metal is soon noticed, and then, with rise in temperature, an increasing evolution of hydrogen followed by a slower evolution, which is long continued, even on application of external heat. A large amount of aluminum is dissolved, enough, in some cases, to make the solution pasty or even solid with the product of the reaction. It has been found that sublimed ferric chloride will also, to a slight degree, bring about the action of aluminum on alcohol, and that even aluminum chloride produces a slight evolution of hydrogen when the chloride is freshly prepared by action of dry hydrochloric acid on metallic aluminum. External application of heat is necessary to the progress of any considerable reaction in the case of ferric chloride or aluminum chloride.

By passing dry hydrochloric acid gas into absolute alcohol standing over chipped aluminum, the metal is readily dissolved with evolution of hydrogen and development of heat. So much aluminum may dissolve in this way that on allowing the solution to cool it becomes solid by separation of a crystalline compound—probably an addition-product of alcohol and aluminum chloride. If, instead of passing into the alcohol a continuous stream of hydrochloric acid gas, a small amount of an alcoholic solution of the gas is added to the alcohol, the action is slower to begin, but increases in vigour till its rate is quite comparable with that induced by the use of anhydrous chlorides of the metals. This form of the reaction is very nicely shown by letting hydrochloric acid gas bubble into the alcohol standing over the chipped aluminum, but removing the gas delivery tube as soon as a fairly rapid evolution of hydrogen is noticed. The reaction will then continue at a good rate, if the mixture is slightly heated occasionally, for more than an hour, until enough aluminum has dissolved to make a jelly when mixed with an equal quantity of water. This hydroxide or basic chloride is, however, entirely soluble in a large quantity of water.

In all the cases noted aluminum chloride has been added, or the conditions were such that it could be formed by decomposition of the hydrochloric acid or anhydrous chloride present. In the case of some of the alcohols it was found, when the stannic chloride was

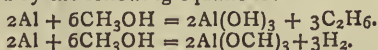
dissolved in a small portion of alcohol, and this solution then added to the main portion of the alcohol, that the action on the aluminum was more vigorous than when the same amount of the chloride was added to the whole of the alcohol. To explain this, it seems to us that in adding the stannic chloride to the smaller quantity of alcohol the temperature was raised high enough to cause the formation of an addition-product of the alcohol and stannic chloride, and that it was upon this compound that the aluminum acted first, and that later it acted on an addition-product of the alcohol with the aluminum chloride which had been found. It is known that all of the anhydrous chlorides used form addition-products with common alcohol. Such addition-products are also known with other alcohols, and it may be said that we obtained a crystalline compound by action of stannic chloride on isopropyl alcohol—probably $\text{SnCl}_4 \cdot 3\text{C}_3\text{H}_7\text{OH}$ as shown by analysis.

For a rapid and satisfactory solution of the aluminum, it may be desirable to have a "couple" of the more easily reducible metals and the aluminum, though our later experiments with hydrochloric acid gas seem to show that it is not necessary.

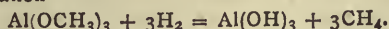
With the reactions involving the use of stannic chloride and hydrochloric acid gas, it is also necessary to have complete dehydration. When the reaction is in full career, the addition of a little water will nearly or entirely stop the action. In this respect there is another marked contrast with the reactions involving mercuric chloride where the presence of water seems to work no harm.

The observations made would then seem to show that, in general, it is necessary to a satisfactory action of aluminum on alcohol, that it should be anhydrous, that it should contain an anhydrous chloride with which it can form an addition-product, and that the aluminum should be coupled with a more easily reducible metal.

Aluminum Methylate.—The methyl alcohol used was dehydrated by anhydrous copper sulphate, and subsequently distilled. Aluminum acts on methyl alcohol under conditions similar to those favourable to its action on common alcohol. On driving off the excess of alcohol a gelatinous mass remains, but on attempting to distil under diminished pressure the mass does not fuse, but simply crumbles to a black powder and yields no condensable distillate. Only two reactions suggest themselves for the action of aluminum on methyl alcohol. They are expressed by the following equations:—



It was found that the evolved gases, passed through sulphuric acid to retain the alcohol, when mixed with air, exploded violently, and gave no indication of the presence of carbon dioxide when clear lime-water was added to the gaseous residue after the explosion, thus showing the presence of hydrogen only. From this the second equation seems the more probable during the first part of the reaction at least. At a later stage it might be that the nascent hydrogen acted on the methylate previously formed and changed it to the hydroxide, as indicated by the equation—



The following experiments show, however, that this is not the case.

In some cases, if not every case, the aluminum alcoholates are soluble in the alcohol from which they are derived. With this thought in mind, an attempt was made to determine whether aluminum methylate had really been formed and decomposed on heating, or aluminum hydroxide had been the final product of the reaction of the aluminum on the alcohol. After a completion of a reaction with aluminum, methyl alcohol, and stannic chloride, a large excess of methyl alcohol was added, and the whole allowed to stand over night. The liquid was decanted from an insoluble residue, and filtered out of

contact with the air. In measured quantities of this solution, the aluminum and the chlorine were determined to ascertain how much of the aluminum could be accounted for as chloride: 10 c.c. of the filtrate were diluted with water, acidified with hydrochloric acid, and the aluminum precipitated by ammonia. From this hydroxide 0.2245 grm. of aluminum oxide were obtained, equivalent to 0.1189 grm. of aluminum. To determine the chlorine, 10 c.c. of the same solution were diluted with water, acidified with nitric acid, and brought to boiling. On adding silver nitrate a precipitate was formed, but it required persistent and very careful filtration to bring the filtrate to final clearness. The reason for this difficulty in clearing, and the cause of the failure of the silver chloride to become curly, has not yet been found. The silver chloride formed weighed 0.4113 grm., equivalent to 0.0897 grm. of chlorine, an amount corresponding to 0.025 grm. of aluminum as chloride.

Considering the similarity of the reaction with this alcohol to those of the other alcohols, and the fact of the evolution of hydrogen during the reaction rather than a hydrocarbon, the presence of aluminum in solution in a form not the chloride can be most easily explained on the supposition that the reaction is normal, that aluminum methylate is formed, but that it is decomposed on attempting to distil it.

Aluminum Propylate.—The normal propyl alcohol used in these experiments was dehydrated by anhydrous copper sulphate. It boils at 97°. In carrying out one of our experiments, 10 grms. of aluminum and 150 c.c. of alcohol were placed in a retort, and then 2 c.c. of fuming stannic chloride were slowly added. No perceptible effect was produced. On applying heat a slight evolution of gas occurred, but ceased on removing the source of heat. On adding 1 c.c. of the chloride an evolution of gas commenced, which was slow at first, but soon became so violent that external cooling was necessary to prevent boiling over. After this spontaneous action became weak, external heating induced further action, which continued several hours until the mass in the retort became quite viscous. In a subsequent experiment approximately the same amounts of aluminum and of alcohol were used, instead of adding the chloride directly to the whole amount of the alcohol, it was first dissolved in a small quantity of the alcohol and then added: 5 c.c. of stannic chloride were dissolved in 10 c.c. propyl alcohol, and of this solution 2½ c.c. were slowly added to the main bulk of the alcohol already standing on the chipped aluminum. Action did not become noticeable for about ten minutes, but then tin began to be deposited; a slow evolution of hydrogen was noticed, which rapidly increased, and soon became violent, as before. The subsequent treatment of the product of reaction was similar to that used with the ethylate. The second of the two operations described above gave a much larger yield of the propylate on distilling, and it showed less indication of the presence of aluminum chloride by fumes at the commencement of the distillation. When the products were re-distilled at a low pressure, they gave an amber-coloured distillate, sometimes solidifying to a clear mass, which gradually became opaque, and sometimes immediately solidified to an opaque solid of the appearance and consistency of fresh commercial grape-sugar. By fractionating, a product was obtained melting at 65° and boiling under 15 m.m. pressure between 235° and 255°. The first fractions were more yellow than the last fraction, which boiled with approximate constancy at 255°. The aluminum in the part boiling at 255° was determined by dissolving in nitric acid, heating the solution till oxidation was no more apparent, evaporating, and igniting the residue. An amount of oxide, Al_2O_3 , was left, which indicated 14.3 per cent of aluminum. By dissolving in hydrochloric acid and precipitating with ammonia, the percentage of aluminum found was 13.2. There should be in aluminum propylate 13.1 per cent of aluminum. The product, made by another operation and distilling between 257° and 262°

at 20 millimetres pressure, was analysed by combustion. In order to protect it from the moisture of the air, it was melted and drawn into weighed glass bulbs with capillary ends, and these, after cleaning and weighing, were dropped into the combustion-tube. The combustion gave—

	H	..	..	..	Per cent.
	C	..	..	..	10.4
	C	..	..	..	53.4
The theory requires—					
	H	..	..	..	10.2
	C	..	..	..	52.9

Aluminum Isopropylate.—Isopropyl alcohol is acted on by aluminum in presence of stannic chloride with evolution of hydrogen under conditions like those used for the normal alcohol, but with less apparent readiness. On heating the product under diminished pressure, however, no distillation takes place, but a decomposition like that observed with methyl alcohol.

Amyl alcohol yields a volatile aluminum compound, of a yellow colour, and boiling at 291° C. under a pressure of 12 m.m.

By the use of aluminum as a reducing-agent, and stannic chloride, platonic chloride, or mercuric chloride as the sensitising agent, there seems no doubt that reduction may be performed in entire absence of water, when the substance to be reduced can be dissolved in any of the alcohols, in benzene, ether, or any solvent with which alcohol will mix when it is added to evolve hydrogen with the sensitised aluminum. From later experiments it is doubtful whether the presence of the precipitated tin, platinum, or mercury is essential. Commercial aluminum at least, containing slight impurities of carbon and iron, evolves hydrogen quite freely under the conditions indicated above, by starting the action with hydrochloric acid.

I wish to express here my hearty thanks to Mr. O. E. Crooker, and especially to Mr. R. F. Hastreiter, by whom much of the experimental work detailed above has been done in connection with graduating theses in this laboratory.—*American Chemical Journal*, vol. xix., No. 7.

PREPARATION OF TELLURIUM AT SCHEMNITZ.

ACCORDING to a long description recently published by Herr J. Farbaký in the *Zeitschrift für Angewandte Chemie*, the Schemnitz tellurium works, in Hungary, were established in 1891, and at first the metalloid was extracted from the ore by precipitation with zinc; but since 1895 the process of precipitation by means of sulphur dioxide, proposed by Dr. A. Maly, has been exclusively employed. The first stage is to attack the ore by strong sulphuric acid, to which end 350 kilos. of concentrated acid are raised to boiling-point in a cast-iron pot, into which 150 kilos. of Transylvania ore are ladled with continuous stirring. This results in the decomposition of the metallic carbonates, and the solution of lead, copper, and zinc along with the tellurium and a part of the silver, leaving gold and silica in the residue. The action of the acid is assisted by gradual and progressive heat until the mass is of a syrupy consistency, a stage attained usually in about six hours. The mass is next lixiviated with 250 to 300 litres of boiling water containing 10 to 15 per cent of hydrochloric acid, to extract the soluble compounds formed, the acid acting as a precipitant of the dissolved silver and re-dissolving the tellurium hydroxide thrown down during the dilution of the mass. This operation, with continued stirring, lasts for six hours, and on the following day the liquid and solid materials are separated by filtration under pressure, the gold and silver in the residual cake being recovered in the ordinary manner. The filtrate is run into lead-lined wooden precipitating tanks about 1 m. long by 0.50 m. wide, into which a current of gaseous sulphur dioxide

(purchased in a liquid form in strong cylinders) is passed. At the end of twelve hours the green solution turns brown and black flakes of tellurium begin to separate out; and when a sample of the liquor ceases to give a precipitate with a little hydrochloric acid and sodium sulphite, the operation is at an end. The mother-liquor is absorbed by slaked lime and ashes, and roasted, &c., for recovering the other metals present. The six days required for precipitation in the tanks may be abbreviated to one-fourth or even less by increasing the contact between the sulphurous acid and the liquor—by using carboys instead of precipitating tanks.

The product is usually between 72 and 85 per cent pure, the chief impurity being copper, of which about 6 per cent is present, along with about 8 per cent of tellurium oxide. So far it is not apparent how the copper is precipitated, and experiments on this point are in progress; the process, however, compared favourably in this respect with the zinc process, since the crude tellurium obtained by the latter was only about 29 per cent pure, and contained some 13 per cent of lead, 15 per cent of copper, and 12 per cent of antimony. It was hoped by repeated solution, re-precipitation, and washing to obtain a product 94 to 97 per cent pure, but the process, though efficient on a small scale, is unsatisfactory in practice, besides being expensive, so the product is dried carefully at a low temperature to prevent oxidation, and the mass is fused in luted earthenware crucibles and either cast in sticks of 1 to 2 in. in diameter or granulated.—*Engineering and Mining Journal*.

CARBIDE OF CALCIUM.

SINCE the Order in Council of the 26th February, 1897, in virtue of which certain parts of the Petroleum Acts, 1871 to 1881, were applied to carbide of calcium, the question of the expediency of exempting small quantities of this substance from the operation of the Order has occupied the attention of the Home Office, and the Secretary of State having been advised that such exemption might be safely extended to quantities of carbide of calcium not exceeding 5 lbs., when kept in separate substantial hermetically closed metal vessels containing not more than 1 lb. each, an Order in Council was made on the 7th July, 1897, authorising the keeping of not more than 5 lbs. of carbide of calcium, in vessels as above described, without a license; and the original Order of the 26th February has been amended accordingly. The amending Order appeared in the *London Gazette* of the 9th July, 1897.

It is to be observed that where the carbide of calcium is not kept in vessels as above described, no quantity may be kept without a license.

Whitehall, 23rd July, 1897.

PROCEEDINGS OF SOCIETIES.

SOCIÉTÉ D'ENCOURAGEMENT POUR L'INDUSTRIE NATIONALE.

July 9, 1897.

M. MASCART, President, in the chair.

THE President announced the sad death of M. Schützenberger, Member of the Committee of Chemical Arts. The obituary eulogium, written by M. Troost, was read by M. Aimé Girard.

Letters of thanks were announced from the recipients of the medals and prizes bestowed at the last meeting, and several new candidates for membership were nominated.

CHEMICAL AND METALLURGICAL SOCIETY, JOHANNESBURG.

Annual Meeting, June 19, 1897.

Mr. W. R. FELDTMANN presided.

THE Secretary read the Annual Report of the Council. For the year under review the Randt has been passing through an industrial crisis, which has now reached an acute stage. During the past year several members of the council have resigned, and others have been elected to fill their places. Twelve ordinary general meetings have been held, at which papers have been read and discussed. The membership of the Society has been increased by twenty during the year, and now numbers 107. The financial position of the Society is satisfactory.

The President then gave his valedictory Address, in which he pointed out the great success which had attended the formation and growth of the Society since its inception three years ago, in spite of wars or rumours of wars and commercial depression, and he foretold a respected and prosperous old age.

Mr. BUTTERS was then unanimously elected President and Dr. Loevy Vice-President for the ensuing year.

Mr. E. H. JOHNSON then read some "Notes on the Reduction of Zinc-Gold Slimes," in which he describes an improved method of cleaning up, by using an improvised filter-pump, and well washing the slimes with a 10 per cent solution of sulphuric acid to remove the zinc. He finds one pound of dilute acid to one pound of moist slimes gives very good results. Working with dilute acid, and not heating, he was able to get a perfect settlement within an hour after leaving off stirring. The advantages of this method over the old are—the elimination of the zinc without calcination; immunity from "dusting," the slimes being wet throughout except in the final operation; and the saving of time, which need not be more than three or three and a half days from the commencement of the clean up to having the bars of gold in the safe.

NOTICES OF BOOKS.

Reports on the Experiments on the Manuring of Oats, Hay, Turnips, and Potatoes. Glasgow and West of Scotland Technical College. Glasgow, 1897.

EACH experiment reported on has been conducted on a number of farms, and the conclusions are, as a rule, based on the average results: there are a number of conditions and circumstances which cause great differences in various soils, quite apart from their original composition, such as the effect of drainage, underground water, previous ploughings, manurings, &c. On this account some exception has been taken to average results, on the grounds that they are from a soil which does not exist, but, on the other hand, it may be said that a soil which possesses every property does not exist, so that perfectly accurate results cannot be obtained for comparison.

Averages may be misleading; if, for example, ten farms give one result and ten farms give another, the average of the twenty is practically valueless; but at the same time, over a large number of cases and apart from great extremes, we think the average becomes more and more the standard as time goes on.

The experiments on the manuring of oats were carried out on fifteen farms. The object aimed at was to find if it was profitable to apply artificial manure to oats, and it was found, amongst other things, that superphosphate increases the proportion of grain; that farm-yard manure largely increases the crop, but principally in straw; that readily available nitrogenous manures, such as nitrate of soda and sulphate of ammonia, give considerable and profitable increases.

Experiments on the manuring of hay show that neither bone-meal nor slag are much good the first year,—with chloride of potassium added it is beneficial to clover, more so than with superphosphate; nitrate of soda alone gives large increase of grass, but diminishes the clover; a combination of nitrogenous, phosphatic, and potassic manures is the best, and benefits both grass and clover alike, and gives a highly profitable return.

Some experiments on the use of seaweed as a manure for potatoes shows that seaweed gives a crop quite equal in weight to that obtained with an equal weight of dung; at the same time, however, the "seaweed" potatoes, both in the raw and cooked conditions, were not considered to be of so good a quality as those from the dunged plots.

The last experiments on the manuring of turnips showed that in all soils, in the south and west of Scotland, basic slag, however used, was not so effective in producing an increase in the turnip crop as was superphosphate containing the same amount of phosphoric acid. In peaty or mossy soils different results were obtained; slag is then better when sown with drills than when scattered broadcast; phosphatic and potassic manures without nitrogen cannot be relied on, but a combination of the three makes a good turnip manure. These conclusions are based solely on the amount of yield shown by the weight of the crop.

The Prospector's Handbook. A Guide for the Prospector and Traveller in search of Metal-bearing or other Valuable Minerals. By J. W. ANDERSON, M.A. (Cantab.), F.R.G.S. Seventh Edition. Revised and much Enlarged. Pp. 176. London: Crosby Lockwood and Son. 1897.

SINCE the first edition was published, in 1885, several important discoveries and openings up of metal-producing countries have occurred. South Africa and Western Australia are the most important of these, though we are now hearing remarkable stories of the fabulous wealth lying about in the Klondike and Yukon regions of North-West Canada.

The geological lessons learnt in South Africa are of great importance, in that they teach us to prospect with eyes and mind open to new impressions and ideas, instead of only looking out for such indications which old experience has taught us to associate with gold-bearing strata.

Among the additions to the Handbook may be mentioned the reference to aluminium ores. This metal, as is well known, is not found in the free state, but generally in combination with silica, oxygen, and fluorine. Its principal ores, besides corundum, are beauxite and cryolite.

The general arrangement of the book is the same as in the previous editions; it comprises chapters on prospecting; blowpipe testing; the character, description, and occurrence of rocks, minerals, and metallic ores; the assaying and treatment of ores; and lastly surveying. There is an Appendix containing the usual tables we expect to find, and very useful they are sometimes, when miles away from everywhere; and a Glossary of terms used in connection with prospecting, mining, &c. Young prospectors should beware of the Glossary, lest, by learning too much of it, they "give themselves away," and show their inexperience.

Reports of the Government Analyst for British Guiana for 1894-5, 1895-6, and 1896-7. Georgetown, Demerara: C. K. Jardine, Printer to the Government, 1895-6-7.

The work done in the Government Laboratory has increased from 2097 samples in 1894-5 to 2399 samples in 1896-7, and we note that the number of official samples has decreased in that period from 1472 to 1450; but in the intermediate year, 1895-6, there was a large increase

of Government work, viz., 1547 samples. As is to be expected, by far the largest number of samples examined were of sugar-cane, sugars, and articles of food and drink. It is remarkable to observe that during these three years no fewer than 38, 46, and 12 samples respectively were of human viscera, in cases of suspected poisoning. We thought a knife or machete was the favourite instrument for argument in the West Indies. But we are glad to see that the latter year, 1896-7, is the lowest (with 1893 when it was the same) in such cases of suspected poisoning ever recorded in the colony.

Report of the Agricultural Work in the Botanic Gardens for the Years 1893-4-5. British Guiana. Georgetown, Demerara: C. K. Jardine, Printer to the Government. 1897.

THE Report over the past three years has been delayed, with the object of recording the termination of the period of the first set of manurial experiments with sugar-cane. This period came to an end in December, 1895, and, as many experiments overlapped, it was considered undesirable to report at arbitrary intermediate periods.

The first experiments discussed in this Report are those with seedling canes, and, as has been pointed out in a previous Report, it is found to be impossible to form any opinion of the richness of the seedling progeny from the richness of the actual parent, on account of the range of variation being so great, and we are forced to regard the saccharine richness of a seedling equally problematical, as are conjectures beforehand as to its colour and size. It is believed that, in the future, improvement lies in raising year by year new varieties from seed, selecting those of greatest vigour, most abundant field yield, and high saccharine contents, in the hope of obtaining a new variety: the "pedigree" seedlings already obtained by this means show that the experimenters are on the threshold of this advance.

The general deductions as to the action of manures and lime upon four crops of sugar-canes grown upon very heavy clay land may be briefly summarised thus:—

1. That nitrogen in the forms of sulphate of ammonia, nitrate of soda, and dried blood, is without doubt the manurial constituent the supply of which mainly governs the yield of the plant.

2. When applied in quantities capable of supplying not more than 40 lbs. of nitrogen per acre, there was practically no difference in the effects of sulphate of ammonia and nitrate of soda, but the former was cheaper. Dried blood was decidedly inferior. The best results were with one-third nitrate of soda and two-thirds sulphate of ammonia.

3. From 2½ to 3 cwt. of sulphate of ammonia per acre appeared to be the most certainly profitable application of nitrogen.

4. Upon plant canes, superphosphate of lime gave considerable and profitable increase of yield when added to manurings of nitrogen and potash.

5. Mineral phosphates require to be applied in such heavy dressings as to render their use unprofitable.

6. Slag phosphate appears to be a promising source of phosphate for plant canes, instead of superphosphate of lime.

7. The addition of potash has little effect; nitrate of potash instead of nitrate of soda was unsatisfactory.

8. The use of lime resulted in largely increased yields. It is satisfactory to note that the West Indian sugar colonies have not been tamely allowing themselves to be driven out of the sugar market, and the colonies themselves destroyed, by the hostile German Sugar Bounties. The planters and others of the colony of British Guiana, we are informed, make great use of the scientific facilities placed at their disposal by the Government; and we are glad to be able to correct the idea, which is perhaps too widely prevalent, that the tropical colonists of Great Britain take but little interest in the scientific aspect of

their work. Reports such as the one now before us show conclusively that the present-day planters are not content to follow the old methods of a century ago, but are doing their best to help themselves in their almost hopeless struggle against the effects of German Sugar Bounties.

CORRESPONDENCE.

PRECIPITATION OF COPPER BY MAGNESIUM.

To the Editor of the Chemical News.

SIR,—In connection with the subject of the displacement of copper by magnesium in solutions of copper sulphate, the following note may be of some interest.

The action appears to be conditioned by the purity of the copper sulphate.

Specimens of bright magnesium ribbon were immersed in an excess of solution—(1) of ordinary blue vitriol, (2) of re-crystallised copper sulphate, bought as pure, and (3) of a specimen of copper sulphate which had been fractionally re-crystallised by myself four times from 2 kilos. of a sample equal in initial purity to (2).

After immersion for two and a half hours, the second specimen was found to have deposited a very small trace of copper. In the third instance the magnesium was withdrawn apparently as bright as when first immersed. On solution it was found to contain a minute trace of copper.

The action appears to be accelerated as time goes on, but even after four days a considerable amount of magnesium is found in the solid residue.

On the other hand, the purest zinc at my disposal was instantly covered with a deposit of copper in each instance, and the action was found to be completed in twelve hours, no trace of zinc being discoverable in the washed pulverulent copper.

I regret that other scientific work has prevented me from continuing these experiments, but I hope that in the course of the winter one or two of my students will have something to say on the action of purified magnesium on purified copper sulphate solutions.—I am, &c.,

SIDNEY A. SWORN.

Chemical Laboratory,
Municipal Technical School,
Gravesend, July 27, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société Chimique de Paris.

Series 3, Vol. xvii.-xviii., No. 12, June 20, 1897.

M. Freundler has studied the decomposition of pyromucates of the alkaline earths under various conditions. This decomposition gives rise to the formation of a small quantity of furfuran, to a carbide, C_3H_4 , and to oxide of carbon.

M. Tardy has separated carbides from bitter fennel and found many interesting bodies.

M. Ponsot, in thinking over a cryoscopic experiment from the calorimetric point of view, finds a means of detecting whether a cryoscopic method regularly used has systematic errors.

On Yellow Light for Polarimetry.—F. Dupont.—The author finds that a mixture of common salt and tri-basic phosphate of soda, fused together in proportions similar to their molecular weights, answers perfectly and gives excellent results. The mixture melts more easily

than sea salt, does not decrepitate, and gives a remarkably steady yellow light. Polarimetric observations are by its use very easy and exact.

Precipitation of Chloride of Copper by Aluminium.—J. B. Senderens.—A reply to the remarks of M. Tommasi.

On Borate of Lithium.—H. Le Chatelier.—Lithium shows several analogies, both to the alkaline metals and the alkaline earths. The author has endeavoured to find whether its borates, even by their composition, show more precise analogies, considering the great variations observed between one metal and another, in the number and composition of the borates of each.

Basic Salts of Cadmium.—M. Tassilly.—Already inserted.

On Dextro-Licarhol.—Ph. Barbier and G. Léser.—Pure licareol is heated for eight hours at 150° or 160° with its own weight of acetic anhydride. At the end of this time the acetic acid is removed by successive washings, the product of the reaction is dried and rectified *in vacuo* in three fractions: the first, from 50° to 80° , consists principally of a mixture of tetratomic terpenes, $C_{10}H_{16}$; the second, from 80° to 105° , which still contains some unacted upon licareol, is again submitted to the action of acetic anhydride, but under modified conditions; the third, from 105° to 130° , submitted to a minute fractionation *in vacuo*, gives an abundant colourless liquid, of agreeable odour, boiling at 119° to 120° under a pressure of 10 m.m.; and which has the composition of the acetic ether of an alcohol, $C_{10}H_{18}O$.

On some Properties of Caffeine.—E. Tassilly.—Crystals of caffeine from aqueous solution were carefully air-dried for a week; they then gave on analysis the formula $C_8H_{10}N_4O_2 \cdot H_2O$. On heating to 50° — 55° this body commences to lose weight, and on increasing the temperature this loss of weight continues; but the whole of the water of crystallisation is not driven off even at 150° , while the caffeine itself is volatile at this temperature. Experiments showed the caffeine is carried off by water vapour when evaporated on a water-bath, or even when heated up to 110° . Further experiments were made on the action of alkalies on caffeine, and it was found that though magnesia has no action, the same cannot be said of baryta and lime, which decompose it with the production of ammonia.

On an Isomer of Disulphide of Diphenylene.—P. Genvesse.—This body is formed while preparing disulphide of diphenylene by the action of sulphur on benzene, in the presence of chloride of aluminium; it is almost insoluble in all solvents, and takes an emerald-green colour under the influence of sulphuric acid.

Decomposition of Pyromucates of the Alkaline Earths by Heat.—P. Freundler.—This paper deals with furfuran and its preparation; the only practical method of preparing this body is that of Limpricht, who obtained it by the distillation of pyromucate of barium with soda-lime. *In vacuo* the decomposition of pyromucates takes place at a lower temperature, but a small quantity of acetone is also formed; under pressure pyromucates can be heated up to 375° without undergoing any change, but towards 400° it decomposes brusquely.

On the Carbide C_3H_4 , a Secondary Product of the Decomposition of Pyromucate of Barium.—P. Freundler.—This carbide fixes bromine in the cold and gives a small quantity of a dibromide boiling at about 50° *in vacuo*; it is extremely irritating to the eyes. The principal product, however, is a liquid tetrabromide, $C_3H_4Br_4$, boiling at 162° under a pressure of 20 m.m. This latter body has not been obtained quite pure, on account of its hygroscopicity and because it loses hydrobromic gas rapidly at ordinary temperatures.

On some Derivatives of Piperonal.—S. Baude and A. Reychler.—Piperonal easily lends itself to the preparation of a series of derivatives similar to those prepared from anisaldehyde, such as methylenedioxy-cinnamate of

ethyl, methylenedioxyphenylpropionic acid, and methylenedioxyphenylacetylene.

On Amidised Amidines.—Charles Lauth.—The author has prepared two amidines giving similar colours, but they are not very stable under the influence of light.

Intervention of Manganese in the Oxidations brought about by Laccase.—G. Bertrand.—Some time ago the author found, while studying the chemical composition of laccase, that the ash contained a relatively high proportion of manganese, but he did not then estimate it. He has now returned to the subject, and has found as much as 2.5 per cent of manganese present. In the action of the ferment he is of the opinion that manganese cannot be replaced by any other metal, not even by iron.

Estimation of the Oxygen Dissolved in Sea-water.—A. Levy and Felix Marboutin.—Already inserted.

Estimation of Cream of Tartar in Wines.—H. Jay.—Several writers on the subject of the estimation of bitartrate of potash rely more on methods of crystallisation than on precipitation; they assume, in fact, that the former is more exact. The author has carefully investigated the subject, and arrives at the conclusion that the results, intrinsically and directly inexact, obtained by the methods of Berthelot and of Fleuriou, are by their corrections, which are constant, nearer the truth than those obtained by crystallisation, in which he finds the excess is not constant.

On the Yellow Colouring-matter of the Oil in White Wines containing Caramel.—Alberto D'Aguiar and Wenceslau da Silva.—Already inserted.

Commercial Rectification of Organic Products.—E. Barillot.—An interesting paper, which, however, requires the accompanying diagram.

MISCELLANEOUS.

Seventy-seventh Annual Announcement of the Philadelphia College of Pharmacy, 1897.—Previous to 1840 pharmacists were not recognised in pharmacopœial conventions, but in that year the College was invited to co-operate with the Committee on Final Publication and Revision. Since then pharmacists have so improved their position that at the last convention in 1890 they numbered sixteen of the twenty-six members. Since the establishment of the institution 14,661 students have matriculated, and 4416 persons have taken the degree of Graduate in Pharmacy.

Paris International Fire Prevention Congress, 1897.—The terrible catastrophe at the Charity Bazaar in May last has determined a number of influential men to form a committee, and call together an International Congress, to discuss all means to prevent and minimise fire risks in theatres, concert halls, and places of public resort. The date of the congress has not yet been finally decided on, but will be announced shortly. In conjunction with the Congress there is being organised an International Exhibition of all engines, inventions, products, and plans for the prevention and extinguishing of fires. Inventors, manufacturers, and engineers are invited to exhibit their machines and inventions; in this way scientific discussion will be strengthened by practical demonstration. One hundred and fifty Senators, Deputies, Municipal Councillors, and scientific men have already joined the committee, and the British Secretary, Mr. Frederick Hoare, 249½, High Holborn, will be glad to receive any data or useful information bearing upon the business of the Congress. France is decidedly behindhand in this matter, and we doubt if she can get outside help better than in England.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1967.

RECENT PROGRESS OF ALCHEMY IN AMERICA.

Reported by H. CARRINGTON BOLTON, Ph.D.

THE suicide of Dr. James Price, the Fellow of the Royal Society who preferred ignominious death to an investigation of his claim to success in transmuting base metals into silver and gold, was a heavy blow to the pretensions of alchemists in England, for we hear but little of the disciples of Hermes in Great Britain during the hundred years that have elapsed since that tragic event. Of vulgar swindlers, such as the American confidence-men who attempted to cheat the Bond-street jeweller by a pretence of "multiplying" sovereigns, there are occasional examples; and there have been instances of successful imposture practised on the Royal Commissioners of Patents, who have granted patents for "getting gold from wheat" by skimming water-washed straw, and for detecting underground treasures by a certain divining-bottle; but these are exceptional, and need not be chronicled in sketching the progress of alchemy in the nineteenth century.

In the United States of America the claims of alchemists have rarely been seriously considered, the people being free from the fetters of authority and tradition, and possessing a shrewd, calculating spirit, symbolised in the word "Yankee." A smart and educated French chemist, named Paraff, visited America a few years ago, pretending to know a process for converting copper into gold, and for a while he succeeded in transferring gold from the pockets of ignorant persons to his own; but when the courts of Peru required him to conduct a transmutation in their presence the career of this modern Cagliostro came to a disastrous end. Within the last twelve months, however, two claims to success in transmutation, or creation of gold, have been made with such boldness, publicity, and persistency, by persons assuming to have superior scientific qualifications, that it has been impossible to ignore their representations, and in one instance the Director of the United States Mint has been called upon to endorse or condemn the secret process of the inventor.

The first of these claims was announced in August, 1896, by Dr. Stephen H. Emmens, a well-known chemist of New York city, whose name is attached to the high explosive "Emmensite" invented by him. Dr. Emmens is a member of several learned societies, and author of papers on chemistry, electricity, and metallurgy, as well as of several novels and poems. In his "Argentaurum Papers" he presents erudite considerations respecting the Newtonian doctrine of gravitation.

To avoid doing injustice to Dr. Emmens the following account of his discovery is given almost wholly in his own words:—

The Transmutation of Silver into Gold.

"Our work, which converts silver into gold, had its origin in the course of certain investigations which I undertook for the purpose of preparing chemically pure nickel. This was in the year 1892. Commodore Folger, who was then Chief of the Bureau of Ordnance of the United States Navy Department, had forwarded to me for investigation a very remarkable specimen of rustless nickel steel which it was proposed to use as a material for torpedo netting. I found the physical properties of this material to be so extraordinary that I desired to investigate the physical behaviour of a similar alloy made with

absolutely pure iron and pure nickel. In attempting to prepare these pure metals, a certain product was obtained which seemed to differ from anything recorded in the textbooks. The same product was subsequently found when the investigation was extended to the case of metallic cobalt. And, finally, those who were associated with me in the investigation agreed with me in considering that the phenomenon observed afforded indications of the existence of some substance common to the whole of the elements in what is known as Series 4 of Group 8 of the Classification of Chemical Elements, now universally adopted by scientists, in accordance with what is known as the 'Periodic Law of the Elements.' We did not further pursue the particular line of investigation upon which we had set out, because it appeared to us almost self-evident that if we were right in supposing a common substance to be present in any single series of elements the same would hold good for each group.

"And as Group I. of the classification contains the precious metals—gold and silver—it was obvious that our time and attention should be directed to these metals rather than to any others. . . .

"It is, of course, out of the question for me to make public the whole of our knowledge in the matter; but I may without danger to our interests give a general explanation of the work which will be satisfactory to the scientific world.

"Our starting-point, so far as silver and gold were concerned, was afforded by the remarkable discoveries of Mr. Carey Lea with regard to the changes that could, by laboratory methods, be induced in the molecular structure of metallic silver. That gentleman discovered a means of causing silver, while still in a metallic condition, to enter into aqueous solution. In other words, he discovered a method of reducing metallic silver to a condition of extremely minute subdivisions. It was found, as might have been expected by anybody familiar with the periodic law of the elements, that this subdivision of metallic silver was attended by very considerable changes in the physical properties of the substance. The inference was obvious that, if such subdivisions could be pushed a stage further, the silver molecules would become dissociated if they were in themselves of composite structure; and, as all chemists have long been agreed respecting the reality of such composite structure, we felt absolutely sure of our ground.

"Accordingly, when by certain physical methods and by the aid of certain apparatus, we succeeded in bringing about a further subdivision of the silver, we were not surprised to find that the substance obtained differed so far from ordinary silver that it could no longer be regarded as the same elementary substance. It seemed to require a new name and a new chemical symbol. Inasmuch, therefore, as our theory was that this substance was common to both gold and silver, and in reality was the raw material out of which both gold and silver were constructed by the hand of Nature, we named the substance "Argentaurum." We gave it also the chemical symbol "Ar."

"The next step was to ascertain whether this substance could be so treated as to be grouped into molecules of greater density than those of silver. Here the element of personal danger was introduced into our researches, and the success of our work on a commercial scale has yet to be assured by the construction and safe manipulation of new apparatus, in which vast energy will be employed. Working upon the necessarily microscopical scale of our experimental researches, we found that the substance called by us 'Argentaurum' can be aggregated into molecules having a density considerably superior to that of silver molecules, and, we think, identical with that of ordinary gold molecules. Whether we are right as to this or not, the condensed argentaurum presents the appearance and is endowed with the properties of ordinary metallic gold. For example, it is green by transmitted light and yellow by reflected light,—properties which, as

all chemists know, are possessed by gold alone. Its resistance to the action of either nitric or hydrochloric acid alone, and its solution by a mixture of these acids, are also distinguishing properties of pure gold, and of no other yellow metal. Under the microscope it is indistinguishable from ordinary gold."

After some comments on the Periodic Law, Dr. Emmens continues:—

"If Mendelejeff's table be examined, it will be seen that a vacant space exists in the Sub-group of Group I., and that this vacant space stands immediately between silver and gold. Our claim is that the hitherto missing element in question is our argentaurum, which in itself therefore is neither silver nor gold, but which may, by our new physical methods, be converted into gold."

Referring to the question of cost the discoverer says:—"We do not consume any chemicals and other costly materials in our process; what we use is mainly energy in some of its various forms, such as heat, electricity, magnetism, gravity, cohesion, chemical affinity, X rays, and the like." "Our chief source of expense is the time required for bringing about the desired molecular changes." And further discussing the expenses, Dr. Emmens estimates that "one ounce of silver will produce three-quarters of an ounce of gold," and that "we can reckon on a profit of at least three dollars per ounce upon all the silver we employ."

In a pamphlet, published in July of this year, Dr. Emmens states that the process of transforming silver into gold depends largely on mechanical treatment, and hints at "the combined effect of impact and a very low temperature." He now operates on Mexican dollars, and, as substantial proof of his labours in transmutation, he has sold to the United States Assay Office six ingots of an alloy of silver and gold, aggregating in value 954 dollars and 80 cents. These ingots have been sold since April 13th, and have been delivered at the rate of two per month; they varied in weight from 7 ounces to 16½ ounces.

Dr. Emmens remarks in this connection, "The gold-producing work in our Argentaurum laboratory is a case of sheer Mammon-seeking; it is not being carried on for the sake of Science, or in a proselytising spirit; no disciples are desired, and no believers are asked for. I have every confidence that the production of Argentaurum gold will be brought up to 50,000 ounces monthly within a year."

The second claimant for a process of converting base metals into precious ones is Edward C. Brice, of Chicago, who has lived a rather chequered life as an enlisted man in the Army, a member of the Police force of Washington city, a promoter of a scheme for manufacturing a patent brick, and a discoverer of the Philosopher's Stone. According to the newspapers, Brice began his operations about three years ago, in Washington, by interesting some rich and credulous men in a secret process for transmutation; several experiments made in their presence seemed to confirm the promoter's statements, but one of those who was supplying money became suspicious, and thought he detected the introduction of gold-leaf into the crucibles used; and later, by skilful diplomacy, he learned from Mrs. Brice that her husband had been buying much gold-leaf for gilding picture-frames! This unkind exposure seems to have caused a temporary suspension of Brice's operations, but in no wise discouraged him, for he next appears in Chicago, where he pushed his old schemes with great success, securing the financial backing of some of the prominent capitalists of that city, bankers, presidents of trust companies, and railway men. All were impressed with the frank manner and attractive plans of Brice, and they advanced him about five thousand dollars, with which he established a small plant capable of yielding fifteen hundred dollars in gold and silver weekly. Business men of ordinary common sense came to the conclusion that Brice "had either gotten something that is invaluable to any Government—or he is a rank fraud."

Brice himself said to one of these wealthy men:—"We do not want capital, we make our capital here. What we need is Government protection, either in the way of purchase or special legislation; the Government alone can entrust the secret to experts. Our experts would go somewhere to a remote place and make all the gold they wanted as fast as we educated them." When capitalists offered to purchase the secret from Brice he refused, saying the Government alone should have the first option. His claim to obtain gold and silver from chemically pure antimony was examined by Robert W. Hunt, a Chicago chemist, and his favourable report "gave support to the credulous and disconcerted the doubters."

On the 7th of May, 1897, Brice filed an application in the United States Patent Office for a patent on a process for creating gold and silver by operating on lead, tin, antimony, and other base metals. The Patent Office twice refused to grant the claim, on the ground that no practical application of the process had been made; but as Brice continued to press his suit, the officials consented to grant him an experimental demonstration of his methods. Owing to inadequate laboratory facilities in the Patent Office building, application was made to the Secretary of the Treasury for permission to use the complete and spacious laboratory of the Mint Bureau. Accordingly the Secretary of the Treasury requested Mr. R. E. Preston, Director of the Mint, to make a thorough investigation of Brice's process, and three expert assayers were appointed for the purpose. The materials needed for the research were bought of reliable dealers in chemicals by Mr. Preston; they comprised 3 pounds of pure antimony, 2 pounds of rolled sulphur, 1 pound of sheet iron, and some pulverised charcoal. The assay commission made a series of experiments according to Brice's instructions, and three weeks' later submitted a report of much interest and value, showing that commercial antimony contained a very small percentage of gold, which was partially recovered by Brice's process. I am indebted to Mr. R. E. Preston, for a copy of this report, which is subjoined.

Public exposure of Brice's scheme for raising money, in the daily press, followed this official report; he himself expressed dissatisfaction with the work of the Commissioners, and still claims that his plant in Chicago is a financial success. Brice's attorney has entered a protest against the findings of the assayers, and here the matter rests.

The circumstances connected with this claim, the appeal to Government authorities, the trial by Officers of the Mint, and the unfavourable report as well as the persistence of the claimant, almost exactly duplicate the events associated with the name of Theodore Tiffereau, in Paris, about forty years ago; and, like Brice, the Frenchman still maintains his discovery, for as recently as December, 1896, Tiffereau addressed a sealed letter to the Academy of Sciences describing a new process to prove that metals are compounds.

These nineteenth century experiences seem worth recording, if only to show how many features they have in common with those of the mediæval chemists,—secret processes, vagueness of description, an assumption of esoteric knowledge expressed in pseudo-philosophic language, personally-conducted experiments with seeming success, and magnificent financial prospects, yet an imperative present need of gold.

(REPORT).

Washington, D.C., May 22nd, 1897.

Hon. R. E. PRESTON,
Director of the Mint,
Washington, D.C.

SIR,—In accordance with your instructions under date of May 3rd, we met at the Mint Bureau on May 5th, to investigate the claim of Mr. E. C. Brice to a process for

producing or creating silver and gold from the base metals, oxides, &c.

Mr. Brice has applied for a patent on this process, and his application has been twice rejected on the ground that the process is inoperative.

We found that you had purchased from the most reputable chemical dealers the materials asked for by Mr. Brice, in a written memorandum, and that the supplies were labelled "chemically pure," as requested by Mr. Brice. A test made by us showed small but weighable quantities of gold and silver in the so-called "chemically pure" antimony. Other samples were procured from different sources with a like result.

While seeking for pure antimony, we accepted the offer of Mr. Brice that he should supervise and direct a trial of his process upon antimony known to contain small amounts of silver and gold, and that he should conduct an assay of the same antimony for a comparison of results, from his own assay methods with those from his "creative process." His assay, in which he scorified one-half assay ton (one assay ton equals 29·166 grms.) with one-half assay ton of lead, showed the antimony to contain 0·066 ounce of gold and 0·317 ounce of silver per ton.

Mr. Brice now subjected five (5) ounces of this antimony to his creative process. His yield, after treatment, showed gold, 0·084 ounce per ton of antimony, and 0·670 ounce silver per ton of antimony used.

Your Committee followed up this work by making an assay of the same metal, following well-known and approved methods of assaying, with the following results:—Gold, 0·100 ounce per ton; and silver, 1·20 ounces per ton of antimony. A comparison of this result will show that Mr. Brice found by his assay sixty-six per cent (66%) of the gold, and twenty-six and forty-one hundredths per cent (26·41%) of the silver actually present in the materials used.

By his "creative process" he recovered eighty-four per cent (84%) of the gold and fifty-five and eighty-four hundredths per cent (55·84%) of the silver originally present in the materials.

It was judged by the Commission that they were not likely to obtain decisive results so long as—working on materials containing appreciable quantities of silver and gold—comparisons would have to be made between minute quantities of those metals found by assay with those obtained by the creative process of Mr. Brice. These differences might be within the limits of error in working, as the amounts obtained were at best very minute, and at times so minute, indeed, that only the most delicate balance in the Institution barely indicated any weight, although this instrument is sensitive to the 1/250th part of a m.grm. (6/100000 grain).

To eliminate the doubt which might thus arise in the minds of those unfamiliar with the limits of accuracy in assaying, it was decided to obtain, if possible, metals entirely free from gold and silver.

The Preparation of Pure Antimony.

We had found all available samples of metallic antimony to contain minute but appreciable quantities of gold and silver. We undertook the task of preparing antimony, which upon assay should not show even a trace of either of those metals. Various methods to attain this end were considered. Distillation of the metal would be obviously ineffective, since in Mint practice it is well known that small amounts of silver and gold are carried off in the vapours of the volatile metals, such as antimony, zinc, &c. An effort was made to prepare pure metal in the wet way from antimony sulphide, but the operation was probably conducted with too much haste, since the resultant metal contained the seemingly inevitable trace of gold and silver. We then had recourse to the process of Capitaine, which is recommended by Gmelin as furnishing the purest antimony (vol. iv., p. 320, published in full in *Journal Pharm.*, xxv., 516). This process consists essentially in

the reduction of metallic antimony from pure double tartrate of antimony and potash. To prevent the reduction of the potash from excess of carbon present, and to form a more fluid melt from which the antimony could settle more perfectly, we added a sufficient amount of pure potassium nitrate to oxidise the excess of carbon present beyond that necessary for the reduction of the oxide of antimony.

The reguline metal thus obtained, amounting to about 20 ounces, was melted under a covering of potassium nitrate. This operation of fusing with the nitrate was repeated until, besides any possible potassium present, a considerable portion of the antimony had been oxidised.

The metal thus obtained, now only some 18 ounces, was finely powdered and treated to a prolonged boiling in distilled water. Not the faintest alkaline reaction was shown by this treatment. We assured ourselves of the absence of gold and silver by repeated assays, and the metal so prepared was used in our subsequent experiments.

Pure Lead.

Lead is used either as metal or oxide in all assaying processes. It is evident therefore that, in this investigation, lead free from gold and silver was equally desirable. It is well known to chemists and assayers that even the so-called "chemically pure" and specially-prepared leads contain traces of these metals. Becker, in his *Monograph on the Comstock Lode (Geological Survey, 1882)*, describes the difficulty he encountered in using even the purest leads available, since the trace of gold and silver was so unevenly distributed that he could not make an allowance for the same with any assurance of accurate results. After much time and labour expended in an attempt to prepare lead or litharge free from gold and silver, he was forced to use a lead containing approximately 0·07 ounce of silver per ton. All the leads sold by chemical dealers to-day as "chemically pure," though more free than Mr. Becker's lead from gold and silver, are only comparatively so, since they all show a minute bead of those metals if sufficient quantity be taken for the test.

In the hope of producing a lead which should be absolutely free from gold and silver, we procured a quantity of Squibb's "chemically pure" lead acetate. This was dissolved in distilled water,—a few drops of potassium iodide added to produce the most insoluble salt of silver known to us under the circumstances,—and, when thoroughly mixed, a few centimetres of dilute sulphuric acid produced a bulky precipitate of lead sulphate which served the purpose of collecting and carrying down any silver iodide which might have been present. It may not be amiss to mention the fact for the benefit of chemists that this lead sulphate precipitate yielded a notable quantity of silver when reduced.

The acetate solution thus freed as we hoped and supposed from silver, was precipitated by dilute sulphuric acid, the lead sulphate washed with distilled water until free from acid. It was then boiled with a solution of ammonia and ammonium carbonate, until the sulphate was converted into carbonate. This compound, when thoroughly washed so as to be free from ammonia salts, was converted into lead oxide (litharge) by heat. Very much to our surprise, four assay tons of the litharge thus prepared yielded a weighable quantity of gold and silver. In view of the limited time at our disposal, we abandoned further efforts in this direction, and having obtained through Mr. Jacob Eckfeldt, Assayer of the Mint at Philadelphia, a sample of lead, which when two (2) assay tons were used for test, showed no visible bead of silver, we used this lead in our subsequent experiments so long as the limited supply lasted. Having now at our disposal antimony and lead, neither of which showed any silver or gold visible to the eye, when used in reasonable quantity, we proceeded to repeat the "creative process" of Mr. Brice, following strictly his instructions as given by him

in his first exhibition of his process, and as slightly modified by his written communication of May 15th.

For this trial we took two (2) assay tons of our specially prepared antimony, — carried it carefully through each step of the "Brice" process, which necessitated the use of six (6) assay tons of lead for its completion.

The result was entirely negative, no trace of gold or silver being visible.

The experiments thus conducted would seem to dispose effectually of Mr. Brice's claim to any creation of gold and silver. But it was deemed best to invite the claimant with his friends to witness, and he to direct, a repetition of the test. This offer was made, and promptly declined by Mr. Brice when he learned that the result of our test was entirely negative. He, however, insisted on the trial of two other and distinctly different methods, neither of which up to this time had been suggested. We yielded, and were given to understand that these methods were improvements on the original process shown us, and that a much larger production of the precious metals might be expected.

Experiment No. 1.—At the behest of Mr. Brice we weighed out two and one-half assay tons of antimony, and subjected it to treatment under his immediate direction. The result was a regulus weighing three assay tons. Mr. Brice directed that the whole of this regulus should be scorified with nine assay tons of lead, and the final button cupelled. The final result was a minute bead of metal, which, when placed on the delicate balance used, weighed 75/1000 of a milligramme (1/1000 grain).

This bead was treated with nitric acid, and a slight trace of gold remained.

Experiment No. 2.—Two (2) assay tons of antimony were treated by a still different method under the direction of Mr. Brice.

A regulus was obtained weighing four and one-quarter assay tons. This was pulverised and treated by a process in which a new feature was introduced. The resulting ore was divided into two parts, because of the necessity of using two different leads, the remaining six assay tons of that from the Philadelphia Mint serving for only two assay tons of the ore. For the remaining two and one-quarter assay tons we were compelled to use seven assay tons of test lead obtained from Richards and Co., and marked "Strictly free from gold and silver." From neither of these operations comprised in Experiment No. 2 was any visible bead obtained.

The contents of the Richards' lead in silver and gold being an unknown quantity, it was thought prudent to conduct a check assay upon this lead, using seven assay tons for the purpose.

A bead was recovered weighing 25/1000 of a m.grm., which when treated with nitric acid showed a slight trace of gold. Mr. Brice in his operation, using this same lead, failed to recover the silver and gold shown to be present in the materials used by him. The loss of this minute quantity of silver and gold would have no significance but for the statement on the part of Mr. Brice that it was from this variation of his process that he expected his best creative results.

Referring to Experiment No. 1, of May 18th, it should be stated that we subsequently received a further supply of the Philadelphia lead, together with a letter from the firm furnishing it, in which they state that the lead was specially prepared for them, and was the purest known to them. They state that it contains less than 0.02 ounce of silver per ton. An assay made by us upon a large amount of the lead confirms the statement of the Philadelphia firm as to its high degree of purity; yet it contains in nine assay tons sufficient silver and gold to fully account for the minute bead obtained by Mr. Brice in Experiment No. 1 of May 18th.

Conclusion.

During these experiments, which have extended over

some three weeks, and have involved an amount of painstaking labour which we hope has not been entirely wasted, we have seen not the slightest evidence of any "creation" or transmutation.

On the contrary, the claimant failed in every instance to recover the entire amount of silver and gold known to be present in the materials. The claimant seems to have devised a variety of irrational and wasteful methods for recovering a portion of the silver and gold known to metallurgists as being present in many commercial metals, such as antimony and lead.

Respectfully submitted,

(Signed) ANDREW MASON,
Sup't. U.S. Assay Office,
New York.

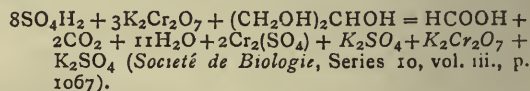
(Signed) D. K. TUTTLE,
Melter and Refiner, U.S. Mint,
Philadelphia.

(Signed) CABELL WHITEHEAD,
Assayer, Bureau of the Mint,
Washington.

ON THE ESTIMATION OF GLYCERIN BY BICHROMATE OF POTASH AND SULPHURIC ACID.

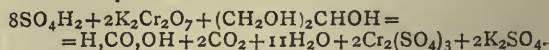
By F. BORDAS and SIG. DE RACZKOWSKI.

WE have given the following equation as the formula for the oxidation of glycerin by bichromate and sulphuric acid:—



Chromic acid oxidises glycerin, giving, in every case, carbonic acid, water, formate of chromium, chromate of chromium, formic aldehyd, &c. In the presence of sulphuric acid there is produced, therefore, besides the carbonic acid and the water, *free formic acid* and sulphate of sesquioxide of chromium.

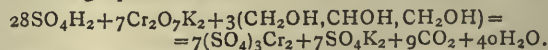
Let us assume the formation of the double salt ($\text{K}_2\text{SO}_4 + \text{K}_2\text{Cr}_2\text{O}_7$) composed of sulphate and bichromate of potash. Now, as this salt will only form when the proportion of sulphuric acid is insufficient to saturate the whole of the potash of the bichromate, we must admit that, as we have an excess of sulphuric acid, the bichromate is entirely decomposed, and therefore this salt cannot be formed under the conditions existing. The amended equation will therefore be—



While in the preceding expression 1 part of glycerin would correspond to 9.62 of bichromate, in the new one it corresponds to only 6.41.

The titration value of the bichromate, of which 1 c.c. should correspond to 1/1000th (the experiment being on 5 c.c. of glycerin solution), which was 48 grms. per litre, now becomes 32 grms. per litre.

In a recent note (*Journ. de Pharm. et de Chim.*, p. 426, May 1, 1897) M. Nicloux asserts that the oxidation of glycerin does not produce formic acid, and he adopts the following equation as the formula of the reaction:—



In this formula the transformation of glycerin into carbonic acid and water is effected *integrally*. He deduces from this that the titration-value of the bichromate solution should be 37.28 per 1000. M. Nicloux then proposes to modify our process by employing a solution of this value.

We could show that M. Nicloux is mistaken in his statements concerning the oxidation of the glycerin by quoting experiments which prove that its decomposition under the influence of bichromate of potash and sulphuric acid *does not proceed integrally*, producing carbonic acid and water, and that there is always undecomposed formic acid present when operating in the way we have described. In fact, the action of chromic acid on formic acid is far from being energetic, seeing that if we heat formic acid with chromic acid, in excess or not, one part decomposes into carbonic acid and water, another part volatilises, and the remainder combines with the sesquioxide of chromium, forming formate of chromium.

It is only after heating for some time in presence of an excess of sulphuric acid, and cooling gradually, that the decomposition is complete.

We prefer, however, not to insist on this point, as discussion is of no interest, given that neither the solution of bichromate of potash at 48 grms. per litre; that of 32 grms. per litre, resulting from the modification of our equation; nor, finally, that of 38 grms. per litre advocated by M. Nicloux, represent definite titration values.

The influence of the proportion of sulphuric acid employed is shown at about one-tenth of a cubic centimetre. It results, therefore, that the titration of the solution of bichromate, being intimately allied to this proportion of sulphuric acid, is absolutely *empirical*, or that the quantity of this solution necessary to obtain the yellowish green tint varies with the quantity of acid, and also with its degree of concentration. This is easily verified by experiment. We can see that, given a solution of bichromate of any titration value, we can determine by successive trials the quantity of acid to use, so that when we use 5 c.c. of glycerin solution, a definite volume of this latter—say, 1 or 2 c.c., for example—will correspond to 18 grms. per litre of glycerin.*

The *modus operandi* proposed by M. Nicloux, which consists of using a solution of 38 grms. per litre in the presence of 4 or even 5 c.c. of pure concentrated sulphuric acid, *boiling if possible*, prevents the grave inconvenience of giving four different results, according as we work with one or the other of the proportions indicated by him, for 4 and 5 c.c. of pure concentrated sulphuric acid, and the same quantities *boiling* constitute four different proportions.

* It can be conceived that there may be under these conditions an unlimited number of modifications in our method of estimation, taking care, of course, to vary in the same manner both the bichromate solution and the quantity of sulphuric acid to be added.

In conclusion, we can only confirm what we have already said (*Comptes Rendus*, Dec. 14, 1896, p. 1072; and *Société de Biologie*, vol. iii., p. 1067), viz., that a solution of pure crystallised bichromate of potash at 24 grms. per litre serves perfectly well; that 2 c.c. of such a solution corresponds to 1 grm. per litre of glycerin, when working on 5 c.c. of glycerin solution and employing *exactly* 2.5 c.c. of pure concentrated sulphuric acid.*

Finally, we would remark that we never pretended to produce a process giving the glycerin to 0.001; for, being colorimetric, there are, of course, difficulties which are inherent to such methods. As we have described it, the process appears to us practicable and able to give results sufficiently near for the purpose for which we devised it; that is to say for the estimation of glycerin in wood ferments.—*Journ. de Pharm. et de Chim.*, Series 6, vol. vi., No. 2.

A NEW AND RAPID METHOD FOR THE

QUALITATIVE SEPARATION OF IRON, ALUMINIUM, CHROMIUM, MANGANESE, ZINC, NICKEL, AND COBALT.

By ALEXANDER RAMSAY CUSHMAN.

Add ammonium chloride, ammonia to alkaline reaction and then ammonium sulphide in slight excess. Warm and filter. Wash the precipitate and remove from the filter while still moist. Warm with moderately dilute hydrochloric acid in a porcelain dish. Complete solution of the precipitate shows the absence of nickel and cobalt. If a black residue remains, one or both are present, in which case add aqua regia, and warm gently until dissolved. Expel excess of acid and free chlorine by evaporation, and then make the solution strongly alkaline with ammonia, after previously adding ammonium chloride in case the amount of hydrochloric acid used upon the sulphides was small. Add bromine solution in excess, and allow to stand a few minutes. Filter and wash.

The Table shows the course of the analysis after this point is reached.—*American Chemical Journal*, vol. xix., No. 7.

* We mentioned 2 c.c. of sulphuric acid as being the proportion to use. Now, with this quantity, 2.1 c.c. of solution of bichromate at 24 grms. per litre is necessary to obtain a yellowish green tint; this would cause an error of 0.05 per 1000.

Precipitate 1.— $\text{Fe}_2\text{O}_3, \text{MnO}_2, \text{H}_2\text{O}, \text{Al}_2(\text{OH})_6, \text{Cr}_2(\text{OH})_6$. Remove from the filter and add KOH in excess, then Br solution and filter.

Filtrate 1.— $\text{ZnCl}_2, \text{NH}_4\text{Cl}, \text{NiCl}_2, \text{NH}_4\text{Cl}, [\text{Cl}.\text{Co}(\text{NH}_3)_5]\text{Cl}_2$. Add KOH in large excess, allow to stand a few minutes and filter.

Residue 2.— $\text{Fe}_2\text{O}_3, \text{MnO}_2, \text{H}_2\text{O}$. Divide in 2 parts. Part 1. Dissolve in HCl, add a few drops of NH_4CNS . Blood red colour shows $\text{Fe}_2(\text{CNS})_6$ and proves Fe.

Part 2. Fuse with $\text{Na}_2\text{CO}_3 + \text{NaNO}_3$ in O. F. of blowpipe. Bluish green mass proves Mn.

Filtrate 2.— $\text{K}_2\text{Al}_2\text{O}_4, \text{K}_2\text{CrO}_4$. Divide in 2 parts. Part 1. Acidify with HCl, add excess of $(\text{NH}_4)_2\text{CO}_3$ and boil. A white flocculent precipitate shows $\text{Al}_2(\text{OH})_6$ and proves Al.

Part 2. Acidify with HCl , H_2O_2 and add $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$. A yellow precipitate shows PbCrO_4 and proves Cr.

Precipitate 3.— $\text{Ni}(\text{OH})_2$. (Greenish white). Confirm in salt of phosphorus bead in the R. F. of the blowpipe. Yellow bead, cold, proves Ni.

Precipitate 4.— $\text{Co}(\text{OH})_3$. Confirm in borax bead, in the O. F. of the blowpipe. Blue bead proves Co.

Filtrate 3.— $[\text{Cl}.\text{Co}(\text{NH}_3)_5]\text{Cl}_2, \text{K}_2\text{O}, \text{ZnO}$. Heat to boiling and filter.

Filtrate 4.—Acidify with HCl , H_2O_2 , and saturate with H_2S . A white, flocculent precipitate shows ZnS. Confirm by ignition with $\text{Co}(\text{NO}_3)_2$ on charcoal. Yellow-green mass proves Zn.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, June 17th, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

Mr. Samuel Pollitt was formally admitted a Fellow of the Society.

A Certificate was read for the first time in favour of Mr. Oscar Guttman, 12, Mark Lane, E.C.

The following were duly elected Fellows of the Society:—William Ackroyd; Walter Harry Barlow; William Malam Brothers; Gerald Noël Brown; Ernest Stuart Cameron; Medwin C. Clutterbuck, B.Sc., Ph.D.; William Cranfield; A. Bilderbeck Gomess; Frederick Roscoe Grundey, B.Sc.; Edward Halliwell; Frank William Harbord; Harold Harman; B. J. Harrington, Ph.D.; John Edwin Mackenzie, B.Sc., Ph.D.; William Robertson Pollock; Lionel Walter K. Scargill, B.A.; James Porter Shenton; William Taverner.

Of the following papers those marked * were read:—

*79. "Molecular Refraction of Dissolved Salts and Acids." Part II. By J. H. GLADSTONE, D.Sc., F.R.S., and W. HIBBERT.

The present paper is a continuation of a previous communication to the Society two years ago, under the same title (*Proc.*, 1895, xi., 120). It is especially concerned in replying to the questions, "Has a salt the same molecular refraction whether it be in the crystalline state or in solution?" and "How far is any refraction change dependent on the solvent used?" The paper also gives some conclusions to which the data appear to lead.

In a table previously published (*Trans.*, 1895, lxvii., 831) there were many comparisons between the specific refraction of solid salts and their value in solution, but no crystals were examined excepting those which had only one axis, or where the different indices were very near together. By adopting the method of Damien as suggested by Pope, we now add seventeen more cases having two or three indices of refraction. The observations are in accordance with the conclusions previously drawn, the refraction of the salt in solution being in some cases greater, and in other cases smaller, than that of the crystallised body. The change of refraction, however, rarely if ever, amounts to 4 per cent.

In making experiments on the effect of different solvents we have examined nine salts and acids, including those published in 1870 (*Trans.*, 1870, xliii., 101). The first result is to show that the specific refraction of the substances dissolved in water does not generally differ much from the value yielded by solution in other solvents. If, however, we examine those substances which show a great change of refraction when deduced from different strengths of solution in water, the result is very striking. A comparison of hydrochloric acid when dissolved in water and in different alcohols and ethers, is shown in a diagram of curves. Whilst in water the specific refraction of the acid is raised in the first instance from about 0.300 to 0.386, and then gradually rises on dilution to 0.400; the acid dissolved in the different alcohols shows a lower starting-point and a more gradual rise, in the following order:—Methyl, ethyl, amyl, and capryl alcohols, followed by a very low starting-point in the case of ethyl ether, with an actual decrease on dilution, and by scarcely any change at all in the case of amyl ether.

In the case of lithium chloride, which gives in water a curve rising on dilution second only to that of hydrochloric acid itself, the solution in alcohol yields a curve very similar in character. Ferric chloride, which is the most striking instance of a great decrease in refraction occurring on dilution with water, shows also a decrease when it is dissolved in alcohol and acetic ether. Nitric acid, when mixed with water, shows a great change of specific re-

fraction, but when dissolved in nitrobenzene shows little if any.

The authors express their growing conviction that neither the salt nor the solvent really changes its specific refraction, but that by their interaction some new product or products result, in quantities determined by the proportionate amount of the two original substances. The action may be one of dissociation or of association, or of some hitherto unrecognised re-distribution. It is the changing proportion of this *tertium quid* which makes itself apparent by the changing specific refraction of the solution.

DISCUSSION.

The PRESIDENT said that the Society was much indebted to Dr. Gladstone for this last addition to his great work on the refractive indices of chemical substances. He wished it were possible to determine directly the refractive index of fluorine, which seemed, from Dr. Gladstone's results with fluorine compounds, to possess the smallest refractive power of any of the elements.

Professor DUNSTAN asked whether the authors had considered the possible influence of the formation of the alkyl chlorides on the solutions of hydrogen chloride in alcohols.

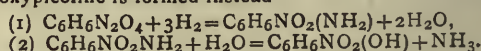
Dr. GLADSTONE, in reply, said they had been unable to detect the formation of any alkyl chloride in the solutions.

*80. "On a Space Formula for Benzene." By J. NORMAN COLLIE, Ph.D., F.R.S. (This paper will appear in our next issue).

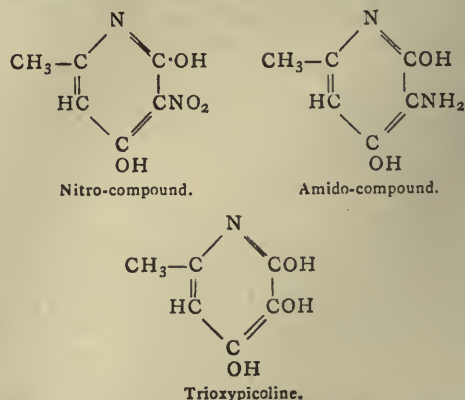
*81. "On the Production of some Nitro- and Amido-Oxypicolines." By A. LAPWORTH, D.Sc., and J. NORMAN COLLIE, Ph.D., F.R.S.

When dioxypicoline (*Trans.*, 1891, 59, 617) is warmed with 60 per cent nitric acid, a nitro-compound is at once produced, $C_6H_7NO_2 + HNO_3 = C_6H_6NO_2 + H_2O$. This nitrodioxypicoline possesses all the properties of a nitrophenol, it is light yellow in colour, and forms salts with bases.

When it is carefully reduced with tin and hydrochloric acid, an amido-oxypicoline results, but should the temperature rise too high a secondary reaction occurs and a trioxypicoline is formed instead—



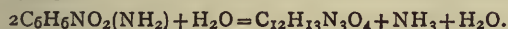
The molecular constitution of these three compounds may be expressed by the formulæ:—



The amido-compound is remarkable for the series of brilliant colours it produces when treated with various oxidising agents; deep indigo-blue with ammonia and air, orange yellow with nitric acid, a deep moss green with alkaline ferricyanide of potassium, and a brilliant magenta with weak acetic acid and potassium bichromate. These colourations are very similar to those produced when various alkaloids are treated in a similar manner.

This amido-oxypicoline forms salts with strong mineral acids but is also capable of liberating carbon dioxide from alkaline carbonates.

When its hydrochloride is only partially neutralised with sodium carbonate and the solution is boiled, an excessively insoluble substance separates, which seems to be a compound of the dioxyamidopicoline and the trioxypicoline—



This insoluble compound by persistent boiling with strong hydrochloric acid is finally changed into the trioxypicoline and ammonia.

The trioxypicoline may be prepared at once from the dioxyamidopicoline hydrochloride, by using the full amount of sodium carbonate necessary to neutralise the hydrochloric acid, and then boiling for half an hour. It then crystallises out in long, needle-shaped crystals.

This trioxypicoline resembles pyrogallol in many of its reactions, it is an excessively powerful reducing agent, and in alkaline solution will develop photographs. It will precipitate silver from a solution containing a considerable quantity of free nitric acid, and it also gives with oxidising agents a series of colour tests similar to those of the dioxyamidopicoline.

*82. "Further Experiments on the Absorption of Moisture by Deliquescent Substances." By H. WILSON HAKE, Ph.D.

In a preliminary note (*Proc.*, 1896, 12, 33) the author showed that certain deliquescent salts, when exposed to the air, attained a maximum of hydration, and that its maximum corresponded to a definite number of molecules in a large number of cases.

In the preliminary experiments no reference was made to the vapour-pressure of water in the air, but in experiments since made, the condition of the atmosphere as regards moisture has been carefully noted, or an artificially saturated atmosphere has been contrived under known conditions of temperature.

Having now experimented with 10 deliquescent chlorides (lithium, magnesium, cadmium, calcium, copper, nickel, cobalt, iron, manganese, and platinum), 3 nitrates (sodium, magnesium, and manganese) with sulphuric acid and with sodium formate, under various conditions, it was found that (1) they attract quantities of water corresponding in all cases to a definite hydrate, (2) after deliquescing to a maximum there followed in all cases a decline in weight, and in four instances the salts returned to their original hydration and crystallised out, and that (3) the amount of hydration, though apparently always corresponding to a definite number of molecules of water, is not always the same, but seems to depend within certain limits both on the temperature and the relative humidity of the atmosphere and also on the conditions under which the air has access to the salt.

The author suggests that the above experiments demonstrate the phenomenon of deliquescence to be caused by hydration of the deliquescent salt.

*83. "The Fusion Point, Boiling Point, and Specific Gravity of Nitro-benzene." By R. J. FRISWELL.

Great discrepancies are found in the books as to the above constants.

Schultz gives in his first edition, 1882, 1'2002 at 0° and 1'1866 at 14'4°; in his second edition, 1'208 at 15°. Fusion point +3°.

Beilstein gives the same, and quotes Mitscherlich for the fusion point and Kopp for the specific gravity.

Gmelin gives 1'209 and +3°, and quotes Mitscherlich. As to boiling point, Gmelin, quoting Mitscherlich, gives 213°. Schultz, 1882, gives 210°; in 1886, 206–207°, Beilstein quoting Städelé, 205° at 730 m.m.

It would thus appear that several of these numbers have been quoted unverified for over 60 years.

No statement of specific gravity of solid nitrobenzene has been published excepting that in *Watts' Dictionary*, given to A. G. Green in a private communication by

R. J. Friswell. Determinations of the various points have been made. Calculated for comparison with water at 4° the sp. grs. are:—

	<i>t</i>	<i>d</i>
Solid	1'5	1'3440
Liquid	3'8	1'2220
"	13'0	1'2160
"	28'0	1'1931

Boiling point corrected 209'0°.

Melting and solidifying point +5°.

Nitrobenzene is remarkable as having a distinctly coloured vapour very closely resembling that of chlorine. The colour is easily visible in a thickness of about 2 inches and is strongly marked when 6–8 inches are examined. The author is not aware of any other organic vapour of so simple a constitution which is visibly coloured.

No bands of absorption are shown in the visible spectrum when light is transmitted through this vapour. The violet and blue are absorbed as with the fluid but less strongly.

*84. "The Action of Light on a Solution of Nitrobenzene in Concentrated Sulphuric Acid." By R. J. FRISWELL.

Nitrobenzene is, as has long been known, readily soluble in concentrated sulphuric acid of 1'84 sp. gr. and upwards, but a comparatively small amount of dilution precipitates it and at about 1'7 the solubility is very slight.

When a solution in pure concentrated acid is exposed to light it slowly darkens. When exposed to direct sunlight, the darkening goes on with great rapidity, and in a few minutes the solution becomes quite black and opaque, then the action ceases. The light from burning magnesium produces the same effect.

The solution has been kept unchanged in the dark for upwards of four years. If the exposure to light takes place in a stoppered bottle, a slight odour of sulphurous acid is perceptible after some time in the air above the solution.

If the nitrobenzene thus used is recovered and re-distilled and re-dissolved in sulphuric acid it behaves in exactly the same way.

Attempts were made to increase the change by spreading the solution on glass beads and between sheets of glass, but the depth of the colour of the product soon brought all change to an end.

Several hundred grammes of the black solution were prepared and attempts made to isolate the products of change, but though a brownish calcium salt was obtained and an ammonium salt in solution, the latter decomposed on evaporation with a caramel like odour; what was left was treated with phosphoric chloride, but no satisfactorily pure product could be obtained: the matter needs further investigation.

The rate of 'blackening' of the solution is undoubtedly a measure of the actinic power of the light.

DISCUSSION.

Mr. A. G. GREEN remarked on the possible similarity between the action of sulphuric acid on nitrobenzene in the presence of light, and the electrolytic reduction of a solution of nitrobenzene in sulphuric acid which had been investigated by Gattermann. In this case it was shown that at first phenylhydroxylamine, then paramidophenol was the product.

Dr. HEWITT said that he had noticed that colouration occurred in sulphonating nitrobenzene, even in the dark.

The PRESIDENT drew attention to the fact that in its changes of density near its solidifying point nitrobenzene appeared to show the same peculiarity as water, viz., an enormous change, but in an opposite direction. The action of light on the nitrobenzene solution in sulphuric acid was remarkable. If a flash of magnesium light were permitted to fall on the nitrobenzene solution through a

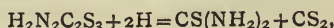
quartz window, he thought that the blackening would furnish a good lecture illustration.

Mr. FRISWELL, in reply, said that he thought it quite possible that the change in the solution of nitrobenzene in sulphuric acid when exposed to light was of the same kind as that induced by electrolysis. He had never observed any colouration on sulphonating nitrobenzene when the materials were pure and light was excluded.

85. "The Reduction of Perthiocyanic Acid." By F. D. CHATTAWAY, M.A., and H. P. STEVENS, B.A.

The atoms forming the molecule of perthiocyanic acid have been assumed to be arranged in a simple ring from the observation that on reduction it yields thiourea and carbon bisulphide. As, however, the properties of the substance render it probable that its molecule is larger than that represented by the simplest possible formula, $\text{H}_2\text{N}_2\text{C}_2\text{S}_3$, the conclusion that in this complex molecule all the structural units have a similar atomic arrangement is only valid if the amounts of thiourea and carbon bisulphide obtained on reduction approach those given by theory. The reduction of perthiocyanic acid has therefore been carefully carried out in various ways to determine the exact amounts of carbon bisulphide and thiourea obtainable and the nature and amount of any other products of the action.

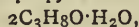
When perthiocyanic acid is reduced by tin and hydrochloric acid, carbon bisulphide and thiourea are produced in almost theoretical amount,



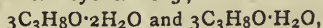
only very small quantities of hydrogen sulphide and carbon dioxide are produced in addition, these being doubtless formed by the hydrolysis of a small portion of the perthiocyanic acid under the influence of the hydrochloric acid, $\text{H}_2\text{N}_2\text{C}_2\text{S}_3 + 2\text{H}_2\text{O} = \text{CS}(\text{NH}_2)_2 + \text{H}_2\text{S} + \text{CO}_2 + \text{S}$.

86. "The So-called Hydrates of Isopropyl Alcohol." By T. E. THORPE, LL.D., F.R.S.

Four hydrates of isopropyl alcohol are stated to exist:—



isolated by Erlenmeyer in 1863;



discovered by Linneman in 1865; and $\text{C}_3\text{H}_8\text{O} \cdot \text{H}_2\text{O}$, prepared by Ruhemann and Carnegie in 1888. All these hydrates boil within a comparatively small range—from 78° to 81° —whereas the amount of water they contain varies from 9 to 23 per cent.

The author gives reasons for doubting the existence of these hydrates as distinct chemical entities capable of definite isolation. By studying the behaviour of mixtures of isopropyl alcohol and water, it would appear that within certain fairly wide limits, water and the alcohol distil together in indefinite proportions, and that the water tends to pass over more rapidly than the alcohol, and hence is found in the largest proportion in the fractions of lowest boiling point. There is no evidence for the existence of these hydrates at their respective boiling points. Nor is there any more evidence for their existence at ordinary temperatures.

By synthetically forming them by the direct addition of the required amount of water to the alcohol, and allowing them to partially evaporate at the ordinary temperature of the air, it is found that the alcoholic strength of the residue is greatly increased; or, in other words, the water evaporates faster than the alcohol, although the latter boils 20° lower than the former. The two substances, therefore, are not in stable union, even at ordinary temperatures.

When the relative densities of the synthetically formed "hydrates" are plotted in terms of the amount of water they contain, the values are found to lie on what is practically a straight line; or, in other words, the density of the mixture is, within the limits studied, very nearly a linear function of the amount of the constituents.

87. "The Carbohydrates of the Cereal Straws." By C. F. CROSS, E. J. BEVAN, and CLAUD SMITH.

This paper deals with the results of further investigations of the products of acid hydrolysis of the cereal straws and of the celluloses isolated from them, including also the closely related esparto-cellulose. The results confirm those previously communicated (*Trans.*, 1896, 69, 804—118), that the furfural-yielding constituents (furfuroids) are selectively attacked and for the most part (90 per cent) dissolved; also from the exceptionally high numbers for cupric reduction, that they must exist in solution in a fully hydrolysed form (monoses).

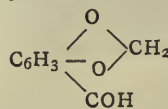
The solutions when neutralised ferment with yeast; carbonic acid and alcohol are produced, and a proportionate effect upon the constants of the solution is shown (density, opticity, cupric reduction, and furfural). Under the conditions, the proportion of the celluloses fermented amounts to 30 per cent of the total dissolved solids. Similar conclusions are deducible from a recent paper by Tollens (*Z. für Landw.*, 1897, 106—107), in which the fate of malt-furfuroids in beer-fermentations is discussed. The experimental numbers in this paper show the disappearance of a large proportion of these constituents in the process.

Since the pentoses entirely resist alcoholic fermentation, as shown by Tollens (*Kohlenhydrate*, ii.), and further confirmed by the authors, as well as by later observations of Tollens privately communicated, it is evident that the group of furfuroids thus fermented is constitutionally distinct from the pentoses.

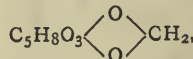
Incidentally to observations on alcoholic fermentation with mixtures of known hexoses and pentoses, the authors find that the latter remain unaffected in presence of hexoses undergoing fermentation. Under certain conditions, however, the pentoses are removed from solution by the yeast organism; the necessary condition appears to be that of "starvation," in the sense, i.e., of the absence of hexoses. The disappearance of the pentose under these conditions is indicated by determinations of furfural and the fall of the furfural numbers. This phenomenon appears to be the simple one of assimilation by the yeast organism, as shown by Bokorny (*Dingl. J.*, 1897, v., 303). The pentose undergoes constitutional change in such assimilation, as the yeast shows no increase in its normal small furfural number.

The authors further discuss the question of the constitution of furfuroids thus shown to yield to alcohol on fermentation, and conclude that the hypotheses of the existence of methylene ethers of the pentoses, or pentose formal, affords, up to the point arrived at, a consistent view of their differentiation from the pentoses.

The history of "piperonal"—



is cited in explanation of the exceptional difficulty of arriving at positive final proof of the analogous constitutional formula—



which sums up the above hypothesis in relation to the group of furfuroids in question. The instability of the pentose as compared with the aromatic residue prevents the application of reactions of resolution (Fittig) or synthesis (Wegscheider) such as have established the methylenic constitution of piperonal.

88. "Studies on the Constitution of Tri-derivatives of Naphthalene No. 16. Conversion of Chloronaphthalene-disulphonic Acids into Dichloronaphthalene-sulphonic Acids." By HENRY E. ARMSTRONG and W. P. WYNNE.

In the course of their studies of naphthalene deriva-

tives, the authors have had occasion to make great use of phosphorus pentachloride as an agent for displacing the SO_3H radicle by chlorine. It was therefore necessary to establish in every possible way the validity of this method of determining constitution in the naphthalene series, as it is obvious that the occurrence of isomeric change in any one case would materially weaken the force of all arguments based on its application.

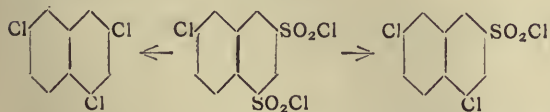
With regard to the nature of the interaction, it is to be noted that in a previous communication (*Proc.*, 1895, xi., 83) it has been shown that it is usually possible to dispense with the pentachloride, and to obtain the chloronaphthalene corresponding with the given sulphonic chloride by merely heating the latter alone under appropriate conditions, but that as a rule the chloronaphthalene is obtained somewhat more readily and in larger relative amount when the pentachloride is used. In other words, the main function of the pentachloride is to promote the elimination of SO_2 from the SO_2Cl radicle.

In the case of the chloronaphthalenesulphonic chlorides, the amount of dichloronaphthalene obtained by means of phosphorus pentachloride is relatively considerable, and the residue left after its removal from the crude product by distillation with steam yields nothing but the original chloronaphthalenesulphonic acid on hydrolysis.

The chloronaphthalenedisulphonic chlorides, however, behave somewhat differently, affording but a comparatively poor yield—rarely exceeding 30 per cent of the theoretical amount—of trichloronaphthalene.

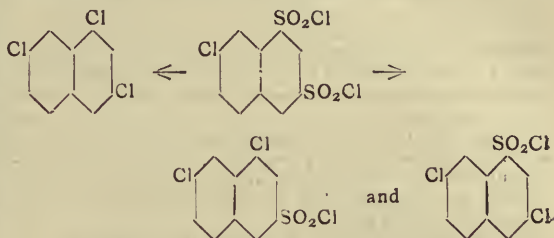
While preparing a full account of their work, the authors have felt it to be incumbent on them to thoroughly examine the residues left after separating the trichloronaphthalenes, which they have had occasion to produce on a large scale (*Proc.*, 1895, xi., 86). Although, in view of the uniformity of the end-products, it was improbable that any change in orientation had taken place at the somewhat high temperatures at which the interactions were effected, it was obviously important to ascertain in every possible way whether such was the fact. The results to be recorded are of interest, as they serve in every case to justify the conclusion previously arrived at, that the treatment of sulphonic chlorides with phosphorus pentachloride may be thoroughly trusted as a means of determining constitution in the naphthalene series. Two cases may be quoted as typical of the behaviour of chloronaphthalene disulphonic chlorides in general.

When 2-chloronaphthalene-4':2'-disulphonic chloride (*Proc.*, 1890, vi., 129) is heated with the theoretical quantity of phosphorus pentachloride at 175° during two hours, it yields both 2:4':2'-trichloronaphthalene and 2:4'-dichloronaphthalene-2'-sulphonic chloride in about equal proportions, about 50 per cent of the material remaining unchanged. 2:4'-Dichloronaphthalene-2'-sulphonic acid affords a sparingly soluble barium salt, crystallising with $3\frac{1}{2}$ molecular proportions of water in microscopic needles; a sparingly soluble potassium salt, containing $1\frac{1}{2}$ molecular proportions, in thin scales; a chloride crystallising from benzene in small prisms melting at 156° ; an amide crystallising from dilute alcohol in slender needles melting at 196° ; and when the chloride is heated at $180-185^\circ$ with phosphorus pentachloride it is converted into 2:4':2'-trichloronaphthalene. On hydrolysing the chloride in sealed tubes with concentrated muriatic acid at 290° , or the potassium salt with a mixture of sulphuric and phosphoric acids and superheated steam, 2:4'-dichloronaphthalene is obtained. The course of change may therefore be thus represented:—



When 2-chloronaphthalene-1':3'-disulphonic chloride (*Proc.*, 1890, vi., 13) is similarly heated with phosphorus

pentachloride, it yields, besides 2:3':1'-trichloronaphthalene, a somewhat larger proportion of a mixture of 2:1'-dichloronaphthalene-3'-sulphonic and 2:3'-dichloronaphthalene-1'-sulphonic (*Proc.*, 1890, vi., 84) chlorides, about 50 per cent of the material remaining unchanged. 2:1'-Dichloronaphthalene-3'-sulphonic acid, the isomeride present in the larger proportion, yields an anhydrous potassium salt, crystallising in thin elongated scales, but exhibiting a tendency to separate in a gelatinous form; a chloride crystallising from benzene and light petroleum in small prisms melting at 130° ; and an amide crystallising from dilute alcohol in slender needles melting at 218° . When the chloride is heated at $180-185^\circ$ with phosphorus pentachloride, 2:1':3'-trichloronaphthalene is formed. On hydrolysing the chloride in sealed tubes with concentrated muriatic acid at 290° , or the potassium salt mixed with sulphuric and phosphoric acids, in superheated steam, 2:1'-dichloronaphthalene is obtained. The course of change may therefore be thus represented:—



The other α - β -disulphonic chlorides behave similarly, the tendency being, however, as in the first of the above instances, to form only one of the two possible isomeric dichloronaphthalenesulphonic chlorides, no doubt because the SO_2 of the SO_2Cl radicle, like the SO_3H radicle, is more easily displaced from α - than from β -positions. It is not certain that these products are intermediate in the strict sense of the term, as the effect of prolonging the heating with phosphorus pentachloride at the minimum temperature at which the reaction takes place serves only to increase the yield both of the dichloro- and trichloro-derivatives. As the dichloronaphthalenesulphonic chlorides produced in these interactions decompose at temperatures a few degrees higher— 10° to 15° in most cases—than those at which the corresponding chloronaphthalenedisulphonic chlorides from which they are obtained undergo change, it is not difficult to understand why they escape attack by phosphorus pentachloride under the conditions observed.

89. "Conversion of 1:1'- into 1:4'-Dichloronaphthalene by Hydrogen Chloride. The Products of Hydrolysis of 1:1'-Dichloronaphthalene-3-sulphonic Acid." By HENRY E. ARMSTRONG and W. P. WYNNE.

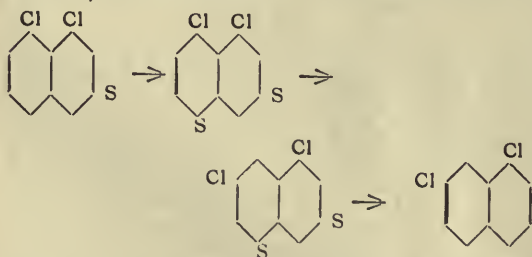
When 1:1'-dichloronaphthalene is heated with concentrated muriatic acid at 290° , it is wholly converted, save for a trace of carbonisation, into the isomeric 1:4'-dichloronaphthalene. This remarkable isomeric change does not seem to occur at temperatures below 200° , but is noticeable at 250° , and complete at 290° ; it does not occur when 1:1'-dichloronaphthalene is heated either alone, or with water, or with concentrated phosphoric acid at 300° , but does happen when it is heated with sulphuric acid of a strength to cause considerable carbonisation. None of the isomeric dichloronaphthalenes show any tendency to change under these conditions.

The experiments which led to these results were made in consequence of the perplexing behaviour of 1:1'-dichloronaphthalene-3-sulphonic acid on hydrolysis. The isomeric α -sulphonic acid (*Proc.*, 1890, vi., 81) requires only a temperature of 230° to effect its hydrolysis, and gives only 1:1'-dichloronaphthalene, whatever be the hydrolytic agent used; the β -sulphonic acid, on the contrary, is not hydrolysed below 285° , and according to the

agent used gives one or other of no less than three dichloronaphthalenes.

1 : 1'-Dichloronaphthalene-3-sulphonic acid is obtained in addition to about an equal proportion of 1 : 1' : 3-trichloronaphthalene when 1-chloronaphthalene-1' : 3-disulphonic chloride (*Proc.*, 1890, vi., 16) is heated with phosphorus pentachloride at 160° (compare preceding abstract). It forms a sparingly soluble anhydrous potassium salt crystallising in thin elongated scales; a chloride crystallising from benzene in thin scales melting at 158°; an amide crystallising from dilute alcohol in short slender needles melting at 197°; and 1 : 1' : 3-trichloronaphthalene when its chloride is heated either with phosphorus pentachloride at 170°, or alone at 200–230°. On hydrolysing the potassium salt with dilute acids, such as 1 per cent sulphuric acid or 50 per cent phosphoric acid at 290°, about 5–10 per cent of the theoretical quantity of 1 : 1'-dichloronaphthalene is obtained, the residue being unchanged salt—a result by which the constitution of the acid is determined beyond doubt. When heated with 5 per cent sulphuric acid or 60 per cent phosphoric acid, carbonisation largely occurs, and with these and stronger acids a small amount of 1 : 4'-dichloronaphthalene is the only substance obtained, a better yield—some 20 per cent of the theoretical—being got when the chloride is heated with concentrated muriatic acid at 290°. The production of 1 : 4' instead of the expected 1 : 1'-dichloronaphthalene under these conditions is to be referred to the action of hydrogen chloride, either present or formed during the carbonisation of the salt.

On effecting hydrolysis by heating the potassium salt, mixed with sulphuric and phosphoric acids, in superheated steam instead of in sealed tubes, an unexpected result was obtained, pure 1 : 2'-dichloronaphthalene, to the extent of 40 per cent of the theoretical amount, being the product, the remainder of the salt being carbonised. The explanation of this change has yet to be given. It is certain that the 1 : 2'-compound is not an intermediate step in the conversion of 1 : 1' into 1 : 4'-dichloronaphthalene during hydrolysis in sealed tubes, both because it is unaffected by prolonged heating with concentrated muriatic acid, and because 1 : 2'-dichloronaphthalene-3-sulphonic acid cannot be detected in the material which has escaped hydrolysis, and, moreover, behaves normally on hydrolysis (compare preceding abstract). It is possible that, under the conditions specified, further sulphonation may precede hydrolysis, and that in consequence of the transference of chlorine to the para-position being thereby prevented, 1 : 2'-dichloronaphthalene is formed, thus:—



Further experiments are being made to test this view.

Of the trichloronaphthalenes the 1 : 2 : 8 modification is the only one which undergoes change when heated with concentrated muriatic acid. Its sulphonic and disulphonic acids behave similarly, but the course of the action has not yet been worked out, owing to want of material.

(To be continued).

Action of Tannin and Gallic Acid upon the Quinoleic Bases.—Oechsner de Coninck.—The bases behave with tannin and gallic acid not only like the pyridic bases, but like pyridic hydrides.—*Comptes Rendus*, cxxv., No. 1.

NOTICES OF BOOKS.

Reform of Chemical and Physical Calculations. By C. J. J. HANSEN, C.E. Printed by the Carlsberg Foundation in Copenhagen. London: E. and F. N. Spon. New York: Spon and Chamberlain. 1897. Pp. 72.

THE deductions contained in this volume are based upon the natural laws of atomic combination, heating, expansion, and compression of æriform substances, and upon the fact discovered by the author—that near the 41° of latitude the specific gravity of oxygen gas of atmospheric density, at the temperature of melting ice, is exactly 1/700th of the gravity of distilled water at its temperature of greatest density.

In different text-books there is great want of uniformity in the values given for many specific gravities, specific heats, &c. (we know one standard work wherein are given no fewer than six values of 1 grm. in grains). This is perhaps because some authors reduce their weights to sea-level, while others take the latitude and elevation of whatever town they may be in. The proposed reform is to reduce all chemical and physical calculations to one common starting-point, from which they can all be fixed by calculation. The proper unit of gravity is hydrogen, but owing to the difficulty in accurately weighing this gas it is better to calculate it from oxygen and nitrogen, both of which have been determined with great accuracy. As all astronomers use the longitude of Greenwich, so, says the author, should all chemists adopt a common circle of latitude, to which all calculations of gravity should be reduced. At a certain latitude, between 41° and 42°, the weight of 1 cubic metre of oxygen at the mean atmospheric pressure and 0° C. is 10/7 kilogramms. This latitude the author proposes to call the *circle of international gravity*. At this latitude the velocity of falling bodies during the first second is 4.90115 metres, and the length of a pendulum making one oscillation per second = 0.993181 metre. A large number of values of various kinds are calculated and given, such as the international atmospheric pressure, boiling-point, specific heats, relative heats, expansion and compression of gases, &c., &c. But there is one important point with which we cannot agree, the author wishes to do away with decimals and go back to the dark ages of vulgar fractions. Some of his calculations done in this manner have a weird and forbidding aspect. Think of it! in adding, subtracting, or multiplying to have to deal with such terminals as 64/185ths, 6/37ths, 2/7ths, 25/37ths, 112/111ths, 867/140ths. These all come in one short calculation of the calories of sensible heat generated by the combustion of 1 kilogram of Dowson's gas.

There is a certain amount of fascination in Mr. Hansen's idea, but we think it is extremely improbable that such a *bouleversement* will be adopted; it cannot be done gradually, and it is certain that even if it were largely adopted, it would not be universal, and we should then have confusion worse confounded.

Electrolytic Quantitative Analysis. ("Quantitative Analyse durch Elektrolyse.") By Dr. ALEXANDER CLASSEN. Fourth Edition, with 74 Illustrations and 6 Plates. Pp. 249. Berlin: Julius Springer. 1897.

THE fourth edition of Dr. Classen's excellent work will be welcomed by chemists. Electricity and chemistry combined are making such rapid strides of late years, as well in the laboratory and assay office as in the metallurgical workshop, that it is only by frequent editions of such books as this that we are enabled to keep *au fait* with what is latest and newest in the methods employed. It is hardly necessary to go through the book *seriatim*; suffice it to say that Dr. Classen begins at the beginning, both with regard to the theoretical and practical side of

the question, and gradually leads the reader up to his own ingenious and refined methods of electro-chemical analysis. The special bits of apparatus he has designed for this work are all described and illustrated, and the best methods of manipulation and procedure with all the metals, and their electrolytic separation one from another, are given at length. Among the six plates at the end of the book we find a contrast between the old purely chemical laboratory and the modern electro-chemical laboratory. Like so many foreign books, it labours under the disadvantage of not having an Index, which is hardly made up for by an excellent table of contents.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 1, July 5, 1897.

Nomination.—In nominating a Foreign Associate *vice* the late M. Tchebicheff, Prof. Virchow obtained an absolute majority of votes (32). Prof. Stokes, the second candidate on the list, received nine votes.

Thermic Mercurial Ammeter.—Charles Carmichel. —The author has the honour of submitting to the Academy novel mercurial ammeters and voltmeters.

New Mercurial Pump without Cocks and Valves. H. Hanriot. —This paper requires the accompanying illustration.

Action of Tellurium Chloride and Fluoride upon the corresponding Hydracids.—R. Metzner.

Reduction of Molybdenum Anhydride by Hydrogen.—M. Guichard. —The reduction of the oxide MoO_3 below 470° is continuous, and leads directly to the intermediate oxides Mo_2O_5 or Mo_5O_{12} . These intermediate oxides cannot be obtained by means of hydrogen. The author promises to give the results of his experiments on the reduction of molybdic anhydride by means of hydrogen at temperatures exceeding 470° .

On Manganomolybdate.—E. Péchard. —There do not seem to exist compounds for the other acids which, like tungstic acid, yield complex acids.

On Veratrylene-Diamine.—Ch. Moureu.

On Paraxylyl-Acetic Acid and Dimethyl-Phenethyl-*oic*-2 Acid.—M. Guerbet.

A Novel Carbohydrate, Carubine.—Jean Effront. —Carubine is widely diffused in nature. We have detected it in oats and barley, and it is probably present in beer.

Fermentations in Compound Mediums of Solid Particles.—Th. Schlösing, jun. —The author concludes that aëration, even without agitation, does not affect the cause of fermentation.

No. 2, July 12, 1897.

The Secretary announced the death of Dr. Steenstrup, a corresponding member of the Section of Anatomy and Zoology, which took place on June 20th.

Election.—M. Goyon was elected a Correspondent for the Section of Rural Economy, *vice* the late M. Hellriegel.

Use of Cupric Salts to Prepare for the Determination of various Elements in Cast-irons and Steels.—MM. Ad. Carnot and Goutal.

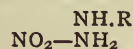
Complexity of the Sheaf of X Rays.—MM. A. Imbert and Bertin-Sans. —A practical conclusion to be drawn from the phenomena observed is, that to obtain a good radiograph, *i.e.*, a proof presenting much contrast, we must

make use of a tube which is still far from being resistant. On the contrary, to effect the radioscopy of a dense medium we must utilise the vacuum at the moment when the less absorbable X rays are present in a sufficient quantity.

On various Basic Copper Salts, and on Brown Cupric Hydrate.—Paul Sabatier.

Reduction of Molybdenum Anhydride by Hydrogen, and the Preparation of Pure Molybdenum.—M. Guichard. —We have established by experiments made at a constant temperature that, in hydrogen between 300° and 470° , the reduction of molybdic anhydride leads to the oxide MoO_2 without passing through the oxides Mo_2O_5 , and Mo_5O_{12} .

Action of Benzoyl Chloride upon the Mono-substituted Orthodiamines.—Fernand Mattalet. —The author undertakes to examine what is the action of benzoyl chloride upon the mono-substituted orthodiamines answering to the general formula—



in which the radicle R may be either fatty or aromatic.

Formation of the Mixed Hydrates of Acetylene and some other Gases.—MM. de Forcrand and Sully Thomas. —This paper is not adapted for useful abstraction.

Action of Sulphuric Acid upon Lavoterebenthenes.—G. Bouchardat and J. Lafont. —Among the liquid products of the reaction there has been found fenchene, $\text{C}_{10}\text{H}_{16}$, derived from fenchol by dehydration.

Development of Aromatic Principles by Fermentation in presence of certain Leaves.—Georges Jacquemin. —To prevent the loss of the volatile aromatic principles the gases of fermentation should be passed through a condenser charged with alcohol.

New Hydrolytic Enzyme—"Caroubinase."—J. Effront. —Caroubinase acts energetically at 40° , and its action increases with the temperature up to 50° . At 70° the action becomes very feeble, and at 80° the enzyme is destroyed.

Optical Analysis of Urine: Diabetic Sugar Thermooptically Positive and Negative.—F. Landalgh.

Composition of Haricots, Lentils, and Peas.—Egyptian lentils, as well as beans of the same origin, are the richest in nitrogen. Those of Auvergne are more nitrogenous than those of Bohemia, Spain, Moravia, and Russia. Immature peas are more nitrogenous than those gathered at full maturity.

Revue Générale des Sciences Pures et Appliquées.

No. 11, June 15, 1897.

On Gas and Petroleum Engines.—Aimé Witz. —This long and interesting article is more suited to an engineering than a chemical paper, but it will well repay perusal by chemical engineers. The history of the gas-engine, which is claimed as a French invention, is traced from the commencement in 1799 to the present time, and a number of illustrations are given.

Liquefaction of Fluorine.—H. Moissan and J. Dewar. —Already inserted in full. In a footnote by the editor the readers are informed that the experiments described in this paper were carried out in the laboratory, and with the resources, of the Royal Institution of Great Britain.

The Dinosauriens.—A. Bigot. —Not suitable for abstraction.

The Cultivation of Cocoa in the French Colonies.—H. Lecompte. —Cocoa was originally found only in Mexico, but has since been introduced into, and found in, many other countries. The largest quantity is produced in Brazil and the English possessions in Central America, where the production is steadily increasing; that in the French colonies remains stationary.

No. 12, June 30, 1897.

This number contains no original matter of chemical interest.

No. 13, June 15, 1897.

On X Rays and Dissociation.—Ch. Guillaume.—The idea of dissociation put forth by Sainte-Claire Deville was familiar to spectroscopists, who found therein an explanation of the identity of the spectra of salts, or of a base, under varying conditions. The author claims that it is proved that dissociation is also caused by X rays. When acting on the flesh the ions cause attenuation of virus, excitation of muscle, and vascularisation. The physiological actions of X rays and of currents of high frequency have not merely a chance analogy, the ultimate cause is the same in either case.

On Cape Diamonds.—L. De Launay.—This paper, which describes the Kimberley Diamond Mines and the mining industry, does not materially differ from the Lecture delivered by Sir William Crookes last year at the Imperial Institute on the same subject, but we can find no acknowledgment of the source.

Applications of Electricity to Artillery.—G. Lavergne.—Not suitable for abstraction in these columns.

MISCELLANEOUS.

Reagents, Reactions, Methods, and Formulæ.—The Editor of the *Pharmaceutical Journal* proposes republishing in book form the list of "Reagents, Reactions, Methods, and Formulæ, known by the Names of their Authors," which has appeared in the *Pharmaceutical Review*, and has since been published as a pamphlet. In order to make the list as complete as possible he will be glad to receive from their authors particulars of any tests or processes associated with the names of any chemists. With the idea of increasing its utility the list is now extended by the addition of particulars regarding a large number of microscopical and bacteriological methods and formulæ from the works of Lee, Squire, Crookshank, and others.

The Yorkshire College, Leeds.—The report of the work of the Textile Industries, Dyeing, and Art Classes of this College, shows that in the Session 1896-7 there has been a slight increase in the number of students, and that satisfactory progress has been made in both the theoretical and experimental work. The teaching is being constantly improved and expanded, to keep up with the developments constantly taking place in the weaving industries. During the last five of six sessions there has been a constant and satisfactory increase of senior students. A twelve horse-power gas-engine has been put down since the last session, and it is now possible to run all the machinery, looms, &c., of the department at one time. It is proposed to add a Carding and Spinning Department, and the draft plans have been passed and are now in the hands of the architect. They will provide ample room for a complete plant of scouring, carding, combing, and spinning machinery for both woollen and worsted yarns, and also suitable rooms for conditioning purposes, and for warping, beaming, and sizing machinery.

Carbide of Calcium and the Petroleum Acts.—In view of the growing importance of carbide of calcium, this substance has, by Order in Council, been brought under the provisions of the Petroleum Acts, 1871 to 1881, and it is now necessary for all users of carbide of calcium to obtain from the local authorities under the Petroleum Acts a license to store it. The Acetylene Illuminating Co., Limited, has been manufacturing carbide of calcium since the spring of 1895, and has always laid the greatest stress on the necessity for proceeding with caution in developing this new industry. When proper precautions are taken, there is no more risk attending the use of acetylene gas than there is with coal-gas. As manufacturers of carbide

of calcium, the above-named company are of opinion that the legislative restrictions will not prove a hardship, and they prefer to see this new industry on a well-defined, instead of on an uncertain, basis. Carbide of calcium in itself is, as is well-known, non-explosive; but when water is added to it, or if it be exposed to a damp atmosphere, acetylene gas is evolved, and if this gas comes in contact with a light an explosion will naturally follow. Its presence is, however, easy to detect on account of its pungent odour. The authorities very properly require that carbide should be "commercially pure"; that is to say, it must not contain any impurities liable to generate siliciuretted or phosphoretted hydrogen, which might render the gas spontaneously explosive. Great stress is laid on the importance of not allowing carbide of calcium or acetylene to come in contact with copper, on account of the ease with which acetylides of copper is formed—this body being highly explosive, a touch being enough to set it off. The Fire Insurance Companies require that carbide, liquid acetylene, or acetylene gas must be stored in a separate building, at least ten feet from any other building, and a safety valve must be fitted to the reservoir or pipes, which will allow the free escape of the gas, outside the building, when the pressure exceeds 4 ozs. to the square inch. We have mentioned a few of the more prominent rules which have been proposed, but they are all in course of revision, it being considered that some are too lax, while others are too stringent; it may, however, be well to emphasize the fact that with ordinary precautions there is no more danger than with coal-gas.

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THE CHEMICAL NEWS.

Vol. LXXVI., No. 1968.

ON THE EXPLANATION OF A PHENOMENON ATTRIBUTED TO A MAGNETIC DEVIATION OF THE X RAYS.

By Sir G. G. STOKES.

IN the *Comptes Rendus* for July 15, 1897 (p. 17), there appeared a note by M. G. de Metz, in which he described an experiment of which the result, according to him, could only be explained by one or the other of these two hypotheses:—Either the X rays are capable of magnetic deviation in an extremely high vacuum, or the cathodic rays are able to traverse the glass envelope of a Crookes tube. I do not think that either of these alternatives contains the true explanation, and I beg the Academy to allow me to submit what is, in my mind, the true explanation of the phenomenon. Everything tends to prove that the X rays are a vibration of the ether, and we may to-day consider it as practically established that this vibration is transversal. If these rays are a vibration of the ether, to suppose that they are capable of magnetic deviation opens up great theoretical difficulties; but, apart from this, I am not aware that such a deviation has ever been demonstrated experimentally. As for the so-called cathodic rays, it seems to me absolutely certain that they are not true rays at all, but simply currents of molecules charged with electricity, thrown off by the cathode. There would be, without doubt, a great difficulty from this point of view, if we were obliged to imagine these molecules capable of passing through the glass of a Crookes tube; the more so, that Crookes himself (*Phil. Trans.*, p. 150, 1879) showed long ago that the cathode rays are stopped by a thin screen of glass, quartz, or mica. But it is in no way necessary to have recourse to this supposition to explain the results obtained by M. de Metz. It seems evident to me that the phenomena which are observed in very high vacua are of the nature of those which have been examined by Spottiswoode and Moulton under the name of *relief-effect* (*Phil. Trans.*, p. 177, 1879). The masses of extremely rarefied air, situated respectively in the Crookes tube and in the cylindrical tube, constitute the two surfaces of a Leyden jar, of which the dielectric is formed by that portion of the envelope of the Crookes tube corresponding with the contour of the cylindrical tube. At each discharge of the induction coil a torrent of negatively electrified molecules is projected against the anti-cathode, or the first surface of the dielectric, which communicates its charge, or a great part of it, either directly to the anode, or, in the first place, to some other part of the internal surface of the Crookes tube. Each momentary charge of the first surface of the dielectric acts inductively on the contents of the cylindrical tube, and produces reciprocally a discharge between the second surface of the dielectric and the aluminium cylinder connected to earth; and in this phase of the reciprocal discharge, where the second surface acts as the cathode, the molecules are projected from this second surface, exactly as from the cathode of the Crookes tube, and they affect a platino-cyanide of barium screen in the same manner.

Although, as I am firmly convinced,—and I believe the greater number of physicists agree with me,—the cathodic rays and the X rays are of a completely different nature, they are both equally capable of affecting a photographic plate or of exciting the fluorescence of a screen of platino-cyanide of barium. This being admitted, the results ob-

tained by M. de Metz are capable of a very simple explanation. When the air in the interior of the cylindrical tube was at the atmospheric pressure, or only at a moderate vacuum, the fluorescence observed was due to the X rays. For, as Lenard has shown (*Wiedermann's Annalen*, li., p. 225, 1894), the cathodic rays—supposing they exist—would be promptly stopped by the air, and would not therefore reach the screen; consequently, the rays producing the fluorescence were found to be insensible to the magnet. On the other hand, at a high vacuum, the cathodic rays, set up by the molecules thrown off by the surface which was made cathodic by induction, were able to reach the screen; and as they were also able to excite a fluorescence much more intense than the X rays, the effect observed was principally due to the cathodic rays; and it is thus that the exciting rays were found to be susceptible of deviation by the magnet.

In offering this explanation, I wish to protect myself against the idea, which might be attributed to me, of explaining in the same manner the apparition of the cathodic rays coming from the second surface of a sheet of aluminium of which the first surface receives the cathodic rays. In this case the *processus* is probably more direct, and presents—to my mind—some analogy with electrolysis.—*Comptes Rendus*, cxxv., No. 4.

ARTIFICIAL LIGHT: MODERN METHODS COMPARED—ELECTRIC. INCANDESCENT, WELSBACH, ACETYLENE.*

By Prof. D. S. JACOBUS.

AT the commencement of this Lecture experiments were made to show the appearance of various colours when under different lights. The two sets of lights to be compared were placed about 6 feet apart, and screens were arranged to shade the audience from the direct glare of the lamps. The colours to be examined were on large sheets of cardboard, bent down the middle to a convenient angle, the apex towards the audience; this being placed between the lamps to be compared, allowed each surface to be illuminated by a different set of lamps. Many colours appeared of a different shade when viewed by the acetylene and the Welsbach lights; the lighter shades of pink especially appeared brighter under the acetylene than under the Welsbach light; but the reverse is the case for some other colours. Ordinary gas, and the incandescent electric light also, produced more lifelike tints when the experiment was made of holding the hand between them and a Welsbach lamp.

In a table of the comparative illuminating-power of water-gas and acetylene-gas, we see that acetylene gives ten times as much light as ordinary illuminative water-gas when burned in a flat-flame burner, and about three times as much when the latter is burned in a Welsbach burner. The percentage of the total heat of combustion of the gas, transformed into light, is also greater for the acetylene than for the ordinary illuminating gas. For a given illumination the atmosphere is vitiated by the carbonic acid formed in burning the acetylene, to a slightly less extent than when burning ordinary illuminating gas in a Welsbach burner.

If 3 per cent of acetylene be mixed with air an explosive mixture is produced. In the case of hydrogen 5 per cent is required, and for coal-gas 8 per cent. The maximum amount of acetylene that can be mixed with air to form an explosive mixture is 82 per cent. It therefore appears that acetylene is more explosive than either hydrogen or coal-gas; but, as the burners used for acetylene would discharge a less amount of gas—say one-fifth that of an

* From a Lecture delivered before the Franklin Institute, March 12th, 1897.

ordinary gas-burner—the accidental opening of a burner would cause the atmosphere of the room, as a whole, to be much less contaminated, in a given time, than with coal-gas. On the other hand, acetylene-gas is so much more nearly of the density of air than ordinary illuminating gas that it will not be diffused as rapidly through the air in case of leakage, and will have a greater tendency to collect in a partially enclosed space, and thus cause an explosion in case the gas were ignited.

According to Berthelot, if gaseous acetylene, under a pressure of about 15 pounds above the atmosphere, or over, be ignited by a spark, or by a heated platinum wire, it will decompose explosively without the presence of air, the explosion becoming more violent as the initial pressure is increased. In exploding, the carbon and hydrogen are dissociated, and the heat absorbed in the original formation is set free. This heat, according to Berthelot, is sufficient to increase the temperature about 5000° F. (provided the pressure be kept constant), or to increase the pressure eleven times if the volume be kept constant.

Berthelot likewise found that liquid acetylene, as transported in tanks under pressure, would explode as readily as the gas. He also made experiments to determine if blows or shocks would cause explosions in cylinders of the liquid, and he found that a shock would not of itself start an explosion, but that when steel cylinders of the liquid acetylene were smashed the blow was usually followed by an explosion, probably produced by the sparks generated by the friction of the pieces of broken steel; the sudden opening of a stop-cock may, he thinks, cause local heating, and thus an explosion.

These investigations show how dangerous acetylene is when stored under pressure greater than that of the atmosphere. That such is the case is borne out by the fact that a number of accidents, accompanied by loss of life, have occurred where it has been used under such conditions.

The cost per hour for the production of 16-candle power of light in New York is as follows:—

Incandescent electric light		1'0 cent.
Illuminating water-gas.	Ordinary 4 ft. burner at 1'25 dollars per 1000 cubic feet.	0'5 "
	Welsbach 1'14 ft. burner at 1'25 dollars per 1000 cub. ft.	0'17 "
Acetylene employed for municipal distribution.	Calcium carbide, converted into gas at 40 dollars per ton, to replace ordinary burners	0'5 "
	Calcium carbide, converted into gas at 19'50 dols. per ton, to replace Welsbach, cost of renewing mantles included	0'17 "

It will be seen from this table that, to compete with an ordinary illuminating water-gas, selling at 1'25 dollars per 1000 feet, the carbide would have to be furnished to the gas-company and converted into gas for 40 dollars per ton, to make the same profit and to be as economical to the consumer as ordinary illuminating gas in a flat-flame burner. To be as economical as ordinary gas burned in Welsbach burners, it would have to be made for 19'50 dollars per ton.

The figures for the acetylene are obtained in the following way:—Assume a plant which is furnishing an illuminating water-gas at 1'25 dollars per 1000 feet. If this plant were converted to an acetylene plant, there are certain expenses—such as cost of distribution, office expenses, &c.—which would remain the same: these constant expenses would be practically all, except the cost of making and storing the gas. To simplify matters, let us consider a plant of, say, 500,000 feet of gas per diem. Then we have—

Paid by consumers per day, 500,000 feet at 1'25 dollars per 1000 feet .. 625'00 dollars.
Cost of gas in holder at 40 cents per 1000 feet 200'00 "

Constant daily expenses, together with profit of gas 425'00 "

To supply the consumers with the same amount of light with the flat-flame burners would require one-tenth the volume of acetylene,—hence 50,000 cubic feet of acetylene would have to be stored in the holder for 200 dollars in order that there might be the same amount of profit for the company. This 50,000 feet would be produced by 5 tons of carbide; hence the cost of 1 ton of carbide, together with making the gas, would have to be 40 dollars to compete with ordinary gas at 1'25 dollars per 1000 feet burned in flat flame burners. In the same way it is calculated that to compete with the Welsbach the calcium carbide must be supplied and converted into gas at about 19'50 dollars per ton.

In some of the earlier literature on acetylene, it was proposed to convert electric-lighting plants into plants for the production of acetylene, and thus obtain economic results. This could not be done economically, for only about half as much light could be obtained from the acetylene as by using the electricity direct for incandescent lamps. The results obtained at Spray show that 2 cubic feet of acetylene could be produced per hour per electrical horse power. This would furnish 80 candle power. If the electricity were used direct with incandescent lamps requiring 4½ watts per candle power, the light produced would be 166 candle power per electrical horse power, or about twice as much light as would be produced by the acetylene. To be as cheap an illuminant, therefore, as electricity, the carbide must be made by some means, say water power, where the cost of power and attendance is much less than at the electric lighting station. It has been shown that for equal illumination the incandescent electric light costs twice as much as gas, when the latter is burned in flat-flame burners. The electric light has, however, held its own against gas on account of its superior qualities; that it has done so at a higher cost to the consumer for a given candle power, is proof that other elements enter into the problem of artificial lighting as strongly as the cost of a given amount of light. From this standpoint it may be argued that acetylene, producing as it does a more brilliant light than any now used for interior lighting, and having the quality of showing the complexion in life-like tints, will have its own field, even should it be the most costly style of illumination.

The whole situation may be summed up by saying that each system of lighting has its own field of usefulness, on account of properties peculiar to itself, which make it more desirable than the others for certain classes of work.

CALCULATION OF THE COEFFICIENTS OF EXPANSION OF GASES FOUNDED ON MY THEORY OF VALENCE.*

By JOACHIM SPERBER.

ACCORDING to Gay-Lussac's law all gases and vapours, *mutatis mutandis*, expand equally for an equal rise of temperature, and indeed for 1° by 0'00366 of their original volume at 0°.

In gases of equal relative heat we may always proceed from volumes in which 1° is at the same time 1 cal.

The relative heat of air, hydrogen, oxygen, and nitrogen is approximately that for which 1 cubic metre is in the mean 0'30726; the relative heats of chlorine and bromine

* A Reprint from the *Zeitschrift Anorg. Chemie.*

vapour are approximately equal, being in the mean 0.38812 per cubic metre in the average.

Hence it follows that in the former gases the volume is 3.254 cubic metres, and in the latter, for 2.576 cubic metres, 1° is at the same time 1 cal.

The author gives the expansion in the case of those elements whose dissociation- and combining-heats have been calculated.

I. *Fluorine*.—In the chemical dissociation of fluorine the amplitude of the fluorine atoms is extended by 0.0266, for which a dissociation-heat of 87.3 cal. per gramme-atom is needed.

II. *Chlorine*.—In the chemical dissociation of chlorine the amplitude of the chlorine atoms is extended by 0.0134 if a dissociation-heat of 44 cal. per gramme-atom is required.

III. *Bromine*.—In the chemical dissociation of bromine the amplitude of the bromine atoms is extended by 0.005, requiring a dissociation-heat of 16.4 cal. per gramme-atom.

IV. *Oxygen*.—In the chemical dissociation of oxygen the amplitude of the oxygen atoms is extended by 0.005, requiring a dissociation-heat of 83.9 cal. per equivalent, or 167.8 calories per gramme-atom.

The expansions of oxygen and chlorine are inversely as the relative heats of these elements.

The expansions of the amplitudes of the atoms of these various elements per calorie coincide up to the sixth decimal and amount to a mean of 0.000304 (6). This number may be suitably named the linear atomistic coefficient of expansion.

Direct determinations have yielded 0.00366, as is well known.

Our computations refer purely to diatomic gases, in which both the atomic weights and the molecular weights are the weights of equal volumes.

FIFTEENTH ANNUAL REPORT OF THE COMMITTEE ON INDEXING CHEMICAL LITERATURE.*

THE Committee on Indexing Chemical Literature presents to the Chemical Section its Fifteenth Annual Report, covering the twelve months ending August, 1897.

Works Published.

"Re-calculation of the Atomic Weights." By Frank Wigglesworth Clarke. New edition, revised and enlarged. Constants of Nature, Part V. Smithsonian Miscellaneous Collections, 1075. City of Washington, 1897. Pp. vi.—370. 8vo.

"Index to the Literature of the Periodic Law." In: "Development of the Periodic Law." By F. P. Venable. Easton, Pa., 1896. 12mo.

"Partial Bibliography of Argon." By C. Le Roy Parker. Accompanying his paper: "Our Present Knowledge of Argon." *J. Am. Chem. Soc.*, xix., 124 (Feb., 1897).

"Bibliography of Agricultural Chemistry (American)." Bulletins of the Office of Experiment Stations, United States Department of Agriculture.

In our Fourteenth Annual Report the following correction should be made: for Bulletin No. 9 read Bulletin No. 19, and add Bulletin No. 27 (1896).

A card-index to Experiment-Station Literature is issued by the Office of Experiment Stations; this is sent *gratis* to all the Agricultural Colleges and Experiment Stations in the United States, and is sold to a limited number of

individuals and institutions. Eleven thousand cards had been distributed prior to September, 1896.

The detailed index included in each volume of the *Experiment Station Record* contains numerous references to chemical articles published by the Experiment Stations in the United States and in foreign countries.

"Abstracts of Chemical Work in Agricultural Science," published in: *Experiment Station Record* issued by the Office of Experiment Stations, United States Department of Agriculture.

These Abstracts were begun in vol. i., No. 1 (September, 1889). Abstracts of foreign investigations were added beginning with vol. ii., No. 8 (March, 1891), and these have been included, with a quite rapid growth in the field covered, up to the present time. The work is in charge of Dr. E. W. Allen, who is assisted by Mr. W. H. Beal in the departments of fertilisers and soils, and by the Committee on Abstracting of the Association of Official Agricultural Chemists.

"The Review of American Chemical Research," edited by Prof. Arthur A. Noyes, began in April, 1895, and formerly published in the *Technology Quarterly*, is continued in the *Journal of the American Chemical Society*.

"Periodicals relating to Chemistry and Physics" in the New York Public Library and Columbia University Library. Bulletin of the New York Public Library Astor, Lenox, and Tilden Foundations. Vol. i., No. 6. June, 1897. Page 152.

A very convenient check-list compiled with bibliographical accuracy, especially useful to students residing in New York and vicinity.

"Bibliography of the Analysis of Chrome-iron Ore, Ferro-chromium, and Chrome-steel." By S. Rideal and S. Rosenblum. *CHEM. NEWS*, vol. lxxiii., p. 2. (Jan. 3, 1896).

"A Bibliography of the Chemistry of Chlorophyll," by L. Marchlewski, accompanies his monograph: "Die Chemie des Chlorophylls." Hamburg and Leipzig. 1895. 8vo.

Reports of Progress.

"A Bibliography of the Metals of the Platinum Group," 1748—1896, by Professor James Lewis Howe, has been completed, and after examination by your Committee has been recommended to the Smithsonian Institution for publication. The work is now going through the press.

"A Review and Bibliography of Metallic Carbides," by Mr. J. A. Mathews, of Columbia University, was submitted to your Committee, and, after examination by each member, the MS. was returned to the author for minor improvements. The suggestions of the Committee were promptly accepted by Mr. Mathews, and the revised work has been recommended to the Smithsonian Institution for publication.

"A Bibliography of Basic Slags, Technical, Analytical, and Agricultural," has been completed by Karl T. McElroy, of the Division of Chemistry, U.S. Department of Agriculture. The channel of publication has not been determined.

The second edition of the "Catalogue of Scientific and Technical Periodicals," 1665—1895, by Dr. H. Carrington Bolton, is entirely printed, but publication is deferred owing to the preparation of a new Library Check List, with which it will be accompanied. The new edition contains 8603 titles.

"A Supplement to the Select Bibliography of Chemistry," 1492—1896, has been completed by Dr. H. Carrington Bolton, who has presented the MS. to the Smithsonian Institution. This Supplement contains about 9000 titles, including many chemical dissertations, and is brought down to the end of the year 1896.

* Advance proofs from the *Proceedings of the American Association for the Advancement of Science*, vol. xlv., 1897. Communicated by H. Carrington Bolton.

Dr. C. H. Joüet reports his "Index to the Literature of Thorium" nearly finished.

Dr. F. W. Traphagen reports "fair progress" on his "Index to the Literature of Tantalum."

Mr. George Wagner reports that he has made progress on the "Bibliography of Oxygen."

Mr. H. E. Brown, under the direction of Professor A. B. Prescott, is preparing a "Bibliography of the Constitution of Morphine and Related Alkaloids."

Professor William Ripley Nichols, of the Massachusetts Institute of Technology, at the time of his death left an unfinished "Index to the Literature of Carbonic Oxide"; the MS. was taken in hand by Professor Augustus H. Gill, of the same institution, who has done considerable work upon it; he now reports that he is not in a position to finish the task, and he is perfectly willing to relinquish the large amount of material accumulated to anyone who would undertake to complete it.

Professor Clement W. Andrews, formerly of the Massachusetts Institute of Technology, and now Librarian of the John Crerar Library, Chicago, reports that he is obliged to abandon work on his "Index to the Literature of Milk," and will be very glad to turn over the material to anyone who cares to undertake to complete the bibliography.

It has always been the aim of the Committee on Indexing Chemical Literature to prevent duplication of work, but failure to inform the Committee of work in progress may defeat this undertaking. An announcement in the Fourteenth Annual Report, of certain work having been nearly completed, surprised a chemist in another part of the country, and has led to the abandonment by the latter of much laborious indexing.

In conclusion, the Committee repeats the statement that it labours to encourage individual enterprise in chemical bibliography, and to record in the annual reports works issued and works in progress.

Address correspondence to the Chairman, at Cosmos Club, Washington, D.C.

Committee:—

H. CARRINGTON BOLTON, Chairman,
F. W. CLARKE,
A. R. LEEDS,
A. B. PRESCOTT,
ALFRED TUCKERMAN,
H. W. WILEY.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, June 17th, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

(Concluded from p. 70).

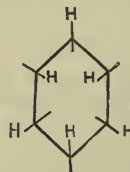
*80. "On a Space Formula for Benzene." By J. NORMAN COLLIE, Ph.D., F.R.S.

In this formula six tetrahedra (to represent the six carbon atoms) are arranged symmetrically in space equidistantly from a common centre, and so that they would occupy the six solid angles of an octahedron. They are connected also symmetrically with one another by single linkings. If the six hydrogen atoms (of benzene) are then arranged symmetrically on these tetrahedra, it will be found that there will be three on one side of the figure and three on the other side.

Movement in this arrangement might take place in two ways; a movement of each tetrahedron about its own

centre, and a movement of each tetrahedron about the centre of gravity of the whole mass.

In the first case, simultaneous rotational movement of each tetrahedron about its centre would bring the combined hydrogen atoms towards the centre of the mass in two distinct sets; those on the 1, 3, 5 carbon atoms and those on the 2, 4, 6 carbon atoms, and a projection of this configuration might be expressed as follows:—



In the second case, movement about the common centre would alter the relative positions of the tetrahedra with regard to one another, bringing into play the six unsaturated points of attraction on these tetrahedra. The projection of these different phases can be represented by the formula given in Fig. 1.

This space formula is therefore in complete accord with that of Kekulé and the centric formula, and shows how they are mutually convertible the one into the other. It also shows how the supposed double bindings in the Kekulé formula shift between the carbon atoms, thus rendering two orthochlorobenzenes impossible. But it differs from both, in that it shows how there may be two distinct sets of hydrogen atoms, and that when one set is inside the molecule, the other set is outside the molecule.

It can offer an explanation also of the fact that when one set of groups is present in a benzenoid compound, further substitution gives ortho- and para-derivatives; whilst, when another set is present, on further substitution meta-derivatives only are formed.

When chlorine adds on nitrobenzene, the chief product is metachloronitrobenzene. The nitro-group belongs to the group that favours the production of meta-derivatives. This nitro-group, being in a sense unsaturated, might possess a certain amount of "residual affinity" which would be sufficient to attract the entering chlorine molecule, and direct it towards the hydrogen atoms that come to the centre at the same moment that it does itself. (See Fig. 2).

On the other hand, in the case of chlorobenzene, if nitric acid be allowed to react with it, no such additive compound would be produced, and the attraction of the three hydrogen atoms attached to the other three carbon atoms might just be sufficient to determine its reaction with them. (See Fig. 3).

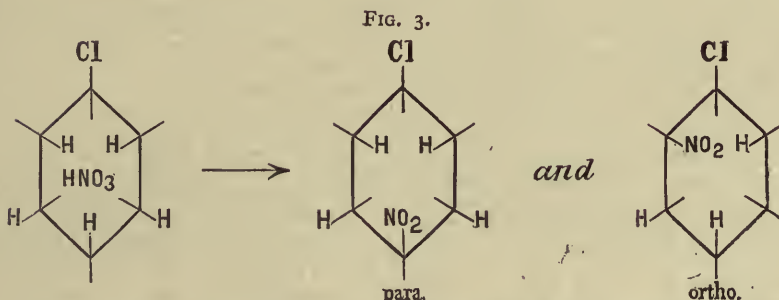
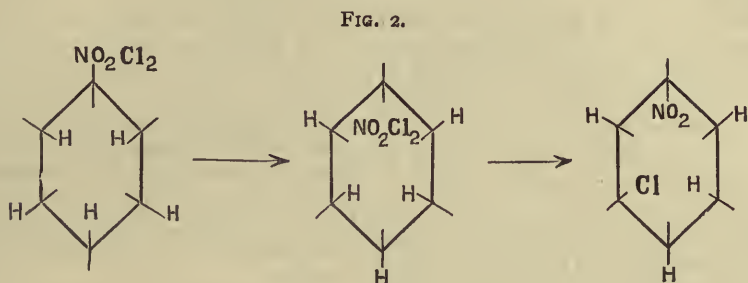
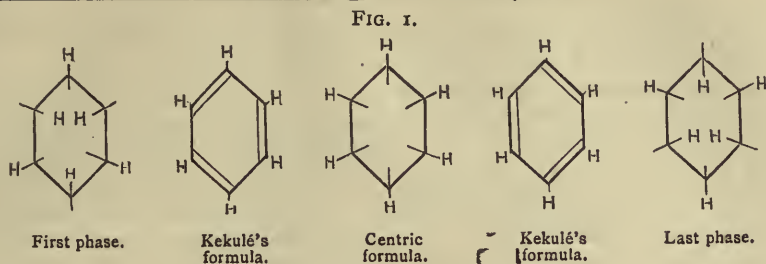
DISCUSSION.

Professor TILDEN thought the paper a valuable contribution to the theory of the construction of the benzene molecule, though he felt doubtful about the validity of the division of the substituents into saturated and unsaturated, since this distinction was, in any case, not a sharp one.

Mr. SWORN considered that the formula proposed did not differ materially from the octahedral formula of Victor Meyer and others, nor did the explanation by the law of substitution now suggested differ substantially from one proposed by himself in a paper published in the *Philosophical Magazine*.

Dr. KIPPING said he was not satisfied with the conclusion that three hydrogen atoms in benzene occupy different positions to the other three. If this were true then two monosubstitution derivatives become possible. From the readiness with which rings are formed from side groups, it would appear that the meta-position corresponds with the ortho-position.

Mr. FRISWELL said that the ordinary nitration of toluene gave a mixture of about 65 parts of orthonitrotoluene and 35 of paranitrotoluene, or very nearly 2 to 1. In conjunction with Dr. T. A. Lawson he had endeavoured



to alter these proportions. Every possible variation of temperature down to nitration at or near zero or as high as 40°, every variation of nitrating mixture from large proportions of sulphuric acid to the use of nitric acid alone, every variation in the order and rate of mixture, the nitration of toluene in which paranitrotoluene had been previously dissolved, had been tried. Yet no important variation of the proportions of the two products had been produced. Mr. A. G. Green had repeated these experiments, and confirmed them. The space formula now suggested by Dr. Collie afforded an explanation, since there were two orthohydrogen atoms during the rotation postulated inside the ring, and only one para-hydrogen atom in that condition. He had long considered this problem, and he had, in conjunction with Mr. C. Mills, commenced to experiment in order to ascertain whether the results might not be due to the existence of two isomeric toluenes.

Professor COLLIE, in reply, pointed out that the space formula for benzene which he had proposed was necessarily similar in many respects to others, notably those of Vaubel and Sachse; but the point on which he wished to lay especial stress was, that there were two sets of hydrogen atoms, and that when one set was inside the molecule the other set was outside.

90. "Note on the Formation of Diacetanilide." By GEORGE YOUNG, Ph D.

The introduction of a second acetyl group into acetanilide has been described in recent years by several authors. Kay (*Ber.*, 1893, xxvi., 2853) treated acetanilide with acetyl chloride at 170–180° from three to four hours. Bistrzycki and Ulfers (*Ber.*, 1894, xxvii., 91)

heated a mixture of acetanilide and acetic anhydride, under pressure, eight to ten hours at 200–205°. Blacher (*Ber.*, 1895, xxviii., 2356) boiled sodio-acetanilide suspended in xylene with acetic anhydride. Tassinari (*Gazz.*, 1894, xxiv., i., 61) acted with acetyl chloride on sodio-acetanilide suspended in benzene. The English and German abstracts of Tassinari's paper state that this author also prepared diacetanilide by treatment of acetanilide with acetic anhydride, but the following quotations from the original paper show that the method used consisted in boiling a mixture of acetanilide, acetic anhydride, and sodium acetate on a reflux apparatus for some hours. In the introduction to his paper, Tassinari makes the general statement, "le diacidanilidi si formano anche con anidride acetica ed acetato sodico a ricadere." It is true that in describing the preparation of diacetanilide, he does not mention sodium acetate—"Trattando dell' acetanilide con anidride acetica, come è detto sopra per la formanilide . . .," but the passage referred to runs: "Scaldando a ricadere per alcune ore un misto di formanilide, anidride acetica, ed acetato sodico. . . ." Further, in a later paper (*Gazz.*, 1894, xxiv., i., 444), in which the work of Bistrzycki and Ulfers is quoted, no notice is taken of a statement by these authors (*loc. cit.*) that, although acetanilide undergoes some change when boiled for two hours with acetic anhydride, they were unable to obtain any pure product from the reaction.

The introduction of the second acetyl group takes place much more easily than might be imagined from the results quoted. If acetanilide be boiled with two to three times its weight of acetic anhydride for half an hour, over 75 per cent is converted into the diacetanilide, which may

be easily purified by the following method:—The cooled product is shaken with benzene and sodium carbonate solution. After drying over calcium chloride, the benzene solution is distilled as far as possible on the water-bath, and the residue treated with light petroleum. The unchanged acetanilide is removed by filtration, and the filtrate evaporated. The residue solidifies on cooling to a crystalline mass, melting at 37–38°. A single recrystallisation, by extraction with cold light petroleum and evaporation of the extract, is sufficient to purify it for analysis, when it melts sharply at 38°.

0.1792 gave 0.4449 CO₂ and 0.1025 H₂O. C=67.71; H=6.35 per cent.

C₆H₅N(COCH₃)₂ requires C=67.79; H=6.21 per cent.

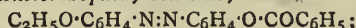
Part of the diacetanilide formed is probably hydrolysed by the treatment with sodium carbonate solution, but by working with not too large quantities, and performing the purification as rapidly as possible, a yield equal to the weight of acetanilide taken may easily be obtained.

91. "Derivatives of Phenetol Azo-phenols. By J. T. HEWITT, M.A., D.Sc., Ph.D., T. S. MOORE, and A. E. PITT.

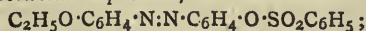
One of the authors has shown (*Ber.*, 1895, xxviii., 799) that certain substitution derivatives of benzenazophenol can form addition products with half a molecule of water, differing very sharply in colour and other physical properties from the corresponding anhydrous compounds. In order to obtain further knowledge of this subject, various series of substituted benzenazophenols are being prepared and examined. This communication deals with ortho- and para-phenol azophenols, C₂H₅O·C₆H₄·N:N·C₆H₄OH, the examination of the meta-derivative being deferred. When a paraoxyazo-compound does form an addition product with water, the addition of the latter can be most frequently brought about by dissolving the azo-compound in benzene and precipitating it by gaseous hydrogen chloride as a hydrochloride of the general formula, X–N:N·C₆H₄OH, HCl, and decomposing this with water. Frequently the molecule of hydrogen chloride is thus replaced by a half molecule of water.

Orthophenetolazophenol was prepared according to the method given by Jacobson and F. Meyer (*Annalen*, 1895, cclxxvii., 213). The melting-point was found to be 128° C. (corr.), Jacobson gives 131° C. The hydrochloride melted between 124° and 129° C. On decomposition with water, the azophenol was regenerated; after air drying it melted at 127–128°. It may be assumed that no water had been added. In order to further characterise the azophenol, the two following derivatives were prepared.

Orthophenetolazophenylbenzoate,—



light scarlet needles, m. p. 98° (corr.). Orthophenetolazophenylbenzene sulphonate,—



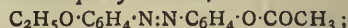
brilliant red needles, m. p. 83° (corr.).

Paraphenetolazophenol has been prepared by Riedel (*D. R. P.*, xlviii., 453), and also by Jacobson and F. Meyer (*Ann.*, 1895, cclxxvii., 215). The former gives the melting-point as 104.5° C., the latter as 125–126°. The method of the latter chemists was used to prepare the compound. The melting-point was found to be 125° (corr.). The hydrochloride gave no very sharp melting-point, beginning to melt at 131°, fusion not being complete until 154°. On treatment with water, a pale yellow powder is obtained melting at about 100°. The same substance may also be obtained by dissolving phenetolazophenol in glacial acetic acid, adding fuming hydrochloric acid, and pouring into water. After careful drying in air the substance melted at 104–109° C. Apparently this substance consists of equimolecular proportions of water and paraphenetolazophenol.

	Calc.	C ₁₄ H ₁₄ N ₂ O ₂ , H ₂ O.	Found.	Mean.
C		64.62	65.10 64.49	64.39 64.68
H		6.15	6.40 6.29	5.95 6.21

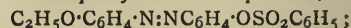
The following derivatives of paraphenetolazophenol have been prepared.

Paraphenetolazophenyl acetate,—



yellow leaflets, m. p. 118° (corr.). Paraphenetolazophenyl benzoate, C₂H₅O·C₆H₄·N:N·C₆H₄·O·COC₆H₅; small reddish brown crystals, m. p. 126° (corr.).

Paraphenetolazophenylbenzene sulphonate,—



large pale yellow plates, m. p. 104° (corr.).

92. "δ-Ketopinic Acid and Camphoic Acid." By W. S. GILLES and F. F. RENWICK.

In the description first given of ketopinic acid (*cf.* Armstrong, *Trans.*, 1896, lxi., 1397), it was stated that the acid was optically inactive, even when prepared by oxidising the most active chlorocamphydrene (pinene hydrochloride) obtainable. As it was a matter of importance to determine whether the inactivity was an inherent property or due to compensation, the authors have applied Pasteur's method, and have succeeded in separating a dextrorotatory modification by fractionally crystallising the mixture of salts obtained by combining the inactive acid with strychnine. δ-Ketopinic acid has the same melting-point as the "inactive" acid from which it is separated.

In their previous note (*Proc.*, 1897, xiv., 64) the authors have stated that when ketopinic acid is oxidised by permanganate, it is converted into a tribasic acid resembling the camphoic acid described by Marsh and Gardner; they are now able to state that the product is camphoic acid, having obtained from it the cis- and trans-camphopyric acids and camphopyric anhydride of these chemists.

An amount of camphoic acid equal to 80 per cent of the weight of the acid oxidised may be obtained by boiling ketopinic acid with a solution containing 50 per cent of nitric acid and adding small quantities of stronger acid (*d*=1.42) from time to time, as the action proceeds.

The authors will endeavour to ascertain if pinophanic and camphoic acids also exist in optically active forms, and what is the behaviour of active ketopinic acid on oxidation. Acids which are probably cis- and trans- forms of pinophanic acid have already been obtained.

93. "Note on Stereoisomeric Di-derivatives of Camphor, and on Nitrocamphor." By T. M. LOWRY, B.Sc.

Having learned from Dr. Armstrong that, in the course of his early studies of camphor derivatives, he had observed that the substances obtained on the one hand by chlorinating bromocamphor and on the other by brominating chlorocamphor are apparently different, the author has submitted the two products to examination.

Brominated chlorocamphor, according to Cazeneuve, melts at 51.5°. The author finds that, on warming a mixture of chlorocamphor and bromine and once crystallising the product from spirit, well-defined crystals are obtained which melt at 53–55°; on analysis, these give results showing them to be bromo-chlorocamphor. By repeated re-crystallisations from a variety of solvents, this product, however, may be resolved into two fractions, alike in composition, but widely different in specific rotatory power. The less soluble product, after being twenty-five times re-crystallised, fused at 61°; its specific rotatory power was [α]_D = 16°. The more soluble fraction—obtained by evaporating the mother liquor, distilling the residue with steam, and re-crystallising the product from dilute spirit—fused at 55°; its specific rotatory power was [α] = 63.9°.

On directly chlorinating bromocamphor, an oil was obtained which could not be caused to crystallise; but a crystalline chlorinated bromocamphor was obtained without difficulty by heating bromocamphor with sulphuryl chloride at 130°. After being twice crystallised from spirit, the product fused at 56°; its specific rotatory power was [α]_D = 25.7° instead of 51°—the value observed in the case of the corresponding product from chlorocamphor. As in the

former case, by repeatedly re-crystallising this product, a less soluble fraction was separated melting at 61.5° , the specific rotatory power of which was $[\alpha]_D = 10.3^{\circ}$; the more soluble fraction from the mother-liquor had the specific rotatory power $[\alpha]_D = 28.3^{\circ}$. There can be no doubt that the two products examined were *isomorphous mixtures* of constituents differing slightly in solubility but widely in specific rotatory power, careful study of the crystalline properties showing the constants to be nearly identical in the two cases. As both yield nothing but ordinary chlorocamphor on reduction, it follows that, in each case, the apparently simple product is a mixture of the two stereoisomeric *aa*-chlorobromocamphors:—



These observations, in fact, undoubtedly afford the proof hitherto wanted, that in the ordinary derivatives of camphor containing halogens, the halogen atoms are both associated with the same carbon atom.

A careful examination both of ordinary dibromocamphor and of nitrobromocamphor has also been made, the result of which is that neither of these is resolvable into isomorphous constituents.

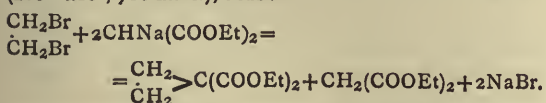
The author finds that when nitrobromocamphor is reduced by means of an alcoholic solution of potash, a nitrocamphor is obtained which has the properties attributed by Cazeneuve to that prepared from nitrochlorocamphor; the substance obtained by R. Schiff must have been impure. By the action of bromine on nitrocamphor dissolved in acetic acid, bromonitrocamphor is reproduced—not the compound $\text{C}_{10}\text{H}_{13}\text{Br}_2\text{N}_2\text{O}_{11}$, which, according to Schiff, is obtained on subjecting the potassium salt of nitrocamphor to the action of bromine.

Nitrocamphor appears to be a birotatory substance, its rotatory power in solution diminishing to a considerable extent as time proceeds—thus, a solution in benzene containing 10 per cent of the substance, gave as initial value $[\alpha]_D = -112.4^{\circ}$, but after three hours -102.7° , and at the expiry of twenty-two hours, when the rotatory power on longer changed, -86.5° .

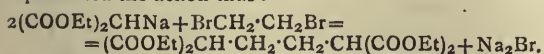
A further observation of interest has been made, viz., that when a solution of nitrocamphor in benzene is evaporated on the water-bath, and the residue is further heated during about an hour, a product is obtained which is less soluble than the original nitrocamphor, and which melts at 190° instead of at 103° . The same substance is formed on heating fused nitrocamphor slightly above its melting-point. The specific rotatory power of this substance in benzene (a 5 per cent solution) is $[\alpha]_D = +187^{\circ}$, and in chloroform $+167^{\circ}$.

94. "The Interaction of Ethylene Dichloride and Ethylic Sodiomalonate." By BEVAN LEAN, D.Sc., B.A., and FREDERIC H. LEES.

It has been shown by Prof. Perkin that, when ethylene dibromide is acted on by ethylic sodiomalonate, the chief product is 1:1-ethylic trimethylene dicarboxylate ($208-210^{\circ}$, 760 m.m.), thus:—

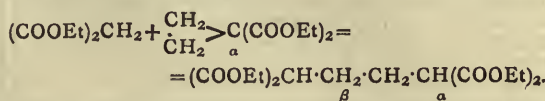


In a later paper, he has shown that a small quantity of an oil of high melting-point is formed, viz., ethylic butanetetracarboxylate (b. p. 240° , 40 m.m.), and he has represented the action thus:—

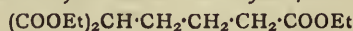


In later papers, he has shown that by substituting ethylene chloride for ethylene bromide the yield of ethylic butanetetracarboxylate can be materially increased, and

it has been proved by Bone and Perkin that the action is represented by the equation—



The authors now show that at the same time small quantities of ethylic butanetetracarboxylate,—



(b. p. $200-205^{\circ}$, 40 m.m.) and ethylic adipate (b. p. 245° , 760 m.m.) are formed. They attribute the formation of these two substances to the action of sodium ethoxide (or perhaps sodium hydroxide, since the materials cannot be entirely free from moisture) upon ethylic butanetetracarboxylate. It cannot be doubted that a similar action is of frequent occurrence, in greater or lesser degree, whenever substances containing two alkyl carboxylic groups attached to one carbon atom are treated with haloid compounds in the presence of sodium ethoxide. The following derivatives have been obtained from ethylic butanetetracarboxylic acid. Ethylic α -ethylbutanetetracarboxylate, $(\text{COOEt})_2\text{CET}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{COOEt}$, a colourless oil, b. p. $200-202^{\circ}$, 32 m.m. Montemartini (*Abstr.*, 1897, lxxii, 19) has also prepared it; he gives the b. p. $205-208^{\circ}$ at 35 m.m. α -Ethylbutanetetracarboxylic acid, $(\text{COOH})_2\text{CET}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{COOH}$, white crystals, m. p. $155-158^{\circ}$. Montemartini describes it as an oil. α -Ethyladipic acid, $\text{COOH}\cdot\text{CHET}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{COOH}$, white crystals, m. p. $48-50^{\circ}$ (Montemartini, 46-49°).

95. "Hexanaphthenes and its Derivatives." Preliminary Note. By EMILY C. FORTEY, B.Sc.

In view of work now being carried on with respect to the naphthenes (Markownikoff, *Ber.*, 1897, xxx., 974, 1211, &c.; Zelinsky, *Ber.*, 1897, xxx., 387, 1532), the author wishes to give a short account of some results, as yet incomplete, on hexanaphthene and its derivatives.

The substance was obtained from American light petroleum by fractional distillation with the aid of a long fractionating column made by Professor Sydney Young, and precisely similar to the one described by him (*CHEMICAL NEWS*, 1895, lxxi, 177). Benzene and toluene were removed by prolonged treatment with a mixture of strong nitric and sulphuric acids. The hydrocarbon finally obtained (after 33 distillations) was not quite free from paraffins; but the purest fraction which boiled at $80.55-80.65^{\circ}$, and had the specific gravity 0.7722 at $0^{\circ}/0^{\circ}$, gave, on analysis, the following result as the mean of three determinations:—C, 85.23; H, 14.60. Calculated for C_6H_{12} :—C, 85.72; H, 14.28. The liquid boiling within 0.4 of a degree was chlorinated by means of a current of chlorine in presence of iron, and a mixture of di-, tri-, and tetrachlorhexanaphthene was obtained. By the action of alcoholic potash on the fraction boiling at $135-140^{\circ}$ under a pressure of 30 m.m., consisting chiefly of trichlorhexanaphthene, hydrochloric acid was eliminated, and the formation of a small quantity of benzene was proved by nitrating it and reducing the nitrobenzene to aniline, which gave the characteristic colouration with bleaching-powder solution. Benzene having thus been obtained from hexanaphthene, the identity of the latter with hexamethylene is no longer questionable.

A small quantity of benzene hydrochloride, $\text{C}_6\text{H}_7\text{Cl}$, was also obtained by the action of alcoholic potash on trichlorhexanaphthene. It boiled at $135-140^{\circ}$, and an analysis gave the following result:—C, 63.20; H, 5.91; Cl, 30.36. Calculated for $\text{C}_6\text{H}_7\text{Cl}$:—C, 62.93; H, 6.12; Cl, 30.95. Hexanaphthene, when heated with fuming nitric acid, was found to be oxidised to adipic acid, as stated by Markownikoff (*Ber.*, 1897, xxx., 975). As both this chemist and Zelinsky appear to have obtained methyl pentamethylene by the action of hydriodic acid on derivatives of hexanaphthene (*Ber.*, 1897, xxx., 387, 1214), it became of interest to see whether the hydrocarbon itself

would undergo isomeric change under similar conditions. Hexanaphthene boiling at 80°C — 80°C was therefore heated in a sealed tube with about five times its volume of hydriodic acid, sp. gr. 1.96, and a little amorphous phosphorus. The tube was heated to about 160° for six hours, from 250° to 270° for three hours, and was maintained at about 250° for four hours longer. The hydrocarbon, after being washed and dried, boiled almost constantly at 80° , showing that it was unchanged hexamethylene.

Zelinsky has found that methylhexamethylene is converted into dimethylpentamethylene by heating with hydriodic acid (*Ber.*, 1897, xxx., 1532); it is therefore interesting to note that hexamethylene itself appears to be much more stable than its derivatives.

It is hoped to obtain a fresh supply of the substance by the fractional distillation of Galician petroleum, when the experiments will be continued.

stearine, which latter substance reduces the melting-point of the mixture below that of either of the ingredients.

We regret to find the un-English orthography "vise" for "vice" adopted in the chapter on the purification of crude oil.

A very important feature of this work is the catalogue of patents and list of patentees. Perhaps we may be permitted to regret the number of patents which form one of the difficulties of the shale-oil trade.

The Analysis of Food and Drugs. Part I.—Milk and Milk-products. By T. H. PEARMAIN and C. G. MOOR, M.A. (Cantab.). Pp. 132. London: Baillière, Tindall, and Cox. 1897.

As this work may run to some 800 or 900 pages, it has been decided to bring it out in several parts, so that each portion may be as complete as possible and contain all the latest information. Each part will be complete in itself, and will have its own index, while a general index will be issued with the last part.

The chief need at the present time is the formation of standards to which the various articles of food should conform, especially in the case of milk, one of the most important and universal foods we have.

It is to the analysis and examination of milk and milk-products that this part is devoted. Milk, which has been described by a well-known doctor as a solid food, is the only article occurring in Nature which combines in the right proportions all the necessary elements requisite to secure proper nutrition, but it is too voluminous to serve as the sole food of adults: it is to prevent this proportion being upset by the interference of the milk-seller that has brought milk analysis into the prominent position it now holds, and renders necessary the small army of inspectors who are constantly on the look-out for adulteration.

For our knowledge of the composition of milk we are indebted, in this country, mainly to the elaborate and prolonged researches of Adams, Bell, Hehner, Stokes, and others. It is found that genuine cow's milk does not normally differ much from the typical analysis here given; it is on the rarest occasions that there is less than 3.5 per cent of fat present, while the average is 4 per cent, and the solids-not-fat may be taken as 8.5 per cent.

It is not surprising that the adulteration of milk to a certain degree is so universal, when in most cases 20 per cent of water, or 30 per cent of skimmed milk, may be added without the resulting mixture falling below the present standard.

The comparison of many thousands of samples a year, for the six years 1890-5, shows a regular steady rate of adulteration of about 12 per cent, and the authors consider that "it is not too much to say that the disgraceful state of the milk trade in this country is fostered, if not actually caused," by the ridiculously low standard adopted by the Laboratory of the Inland Revenue Department, "by which, of course, analysts are compelled to abide, or take the risk of being over-ruled by the referees, who are thought to be infallible in the eyes of some magistrates."

There are several different methods of determining the fat, such as the mechanical methods, and simple extraction with a solvent of the dried milk by Bell's, Adams's, or Macfarlane's method; or extraction of the fat by ether, after destroying the casein by acid. All these methods, as well as those for the estimation of proteids, are fully described in these pages.

The most commonly used preservatives to be found in milk are borax, formaldehyd, salicylic acid, and potassium chromate, and there is no doubt that formaldehyd, or formalin,—by which name it is also known,—being, it is alleged, the most effective, will, when it becomes more generally known, supersede boric acid as a preservative. Two or three drops of formalin in a pint of milk are said to keep it fresh for several days, and the addition of 0.05 per cent should preserve milk for months.

NOTICES OF BOOKS.

A Practical Treatise on Mineral Oils and their By-products; including a Short History of the Scotch Shale Oil Industry, the Geological and Geographical Distribution of Scotch Shales, Recovery of Acid and Soda used in Oil-refining, and a List of Patents relative to Apparatus and Processes for obtaining and refining Mineral Oils. By ILTYD I. REDWOOD, Member of the Society of Chemical Industry (England). London: E. and F. N. Spon, Lim., 125, Strand. New York: Spon and Chamberlain. 1897.

MR. ILTYD I. REDWOOD merits the gratitude of the chemico-technical world for his comprehensive work, the value of which is enhanced by the portrait of the late James Young, the founder of the Scottish mineral oil industry.

The first chapter treats of the history of the trade. James Young was what is called a "self-made man." He became demonstrator and assistant to Professor Graham, and amidst many difficulties laid the foundation of the new manufacture. The famous Bathgate Oil-works were erected in 1851, and after many ups and downs have reached their present position.

Mr. Redwood foretells that unless a unanimous and harmonious combination of the Companies soon takes place, we "shall in a few years be left to mourn the loss of one of Scotland's most important industries."

The true shales are confined to a district extending 12 miles north and south by about 25 miles east and west. They lie in the calciferous sandstone series, lying between the Mountain and the Buriehouse limestones.

Shale is distinguished from coal by the lack of the intense blackness of the latter mineral; it is less easily broken, and then displays a conchoidal fracture.

The proportions of crude oil yielded by the mineral vary in different localities. Thus the Denbrae shale yields 10.90 gallons of crude oil per ton, whilst the true Boghead mineral yields from 85 to 128 gallons and the methyl brown coal from 65 to 90 gallons. A large yield of crude oil is not always a proof of high value, since a high yield of oil is often accompanied by a low percentage of paraffin wax and an abundance of lubricating oils of low specific gravity.

We learn that mineral oils alike in specific gravity often vary greatly in viscosity. Hence consumers prefer to buy oils by the viscosity test rather than by specific gravity. Still it is not safe to rely exclusively upon viscosity. The consumer is therefore recommended to have the cold test and the viscosity of samples both carefully ascertained. The relation between these two sets of data is shown in a table given on p. 221. Further tables (p. 225) show the melting-points of mixtures of wax and

Potassium chromate is not known as a milk preservative in this country, but it is stated to be used on the Continent.

Milk contains, as a rule, a large number of bacteria, for the most part derived from the external conditions and surroundings of the cow; the numbers may even run so high as three or more millions per c.c.; and as many of these, which are capable of causing disease, do so by producing toxic decomposition products, which is greatly increased by a rise of temperature, it is essential that the very greatest care should be taken to keep the temperature as low as possible, from the time when the milk is drawn to the time when it is consumed or cooked. There is little doubt that the great mortality among young children from intestinal tuberculosis, is due to the presence of the tubercle bacillus in the milk used.

The sterilisation of milk is a most important subject, and we are sorry to see that it is very lightly passed over in this volume; it is of course very necessary to have everything connected with the milk supply scrupulously clean, but it is also necessary to have some regular recognised process by which the ubiquitous *Bacillus tuberculosis* can be killed instead of remaining to breed in the milk. Pasteurisation is coming widely into use on the Continent, and we should be glad to hear of its general adoption in this country.

The authors are to be congratulated on having produced a most useful and readable book, and we can only hope the parts yet to come will be worthy of Part I.

Monopolies by Patents, and the Statutable Remedies available to the Public. By J. W. GORDON, of the Middle Temple, Barrister-at-Law. Pp. 300. London: Stevens and Sons, Lim. 1897.

IN his Preface the author explains his reason for taking the word "Monopoly" for the purpose of the title of this book. The word Monopoly, as now generally accepted, no longer has the meaning in law that it had in the time of Blackburn; want of a better one is the reason of the present title used in the now-forgotten sense which it formerly had, a sense in which it implies that not a single individual must be prevented from freedom to follow any lawful trade.

The Patent Law took its rise from the time of Queen Elizabeth, when, owing to the interference caused to lawful trade by the large number of monopolies she had granted, a reaction was produced in the public mind, which might have resulted in a political convulsion had she not afforded some relief by recalling a few of the most iniquitous of the monopolies she had granted.

The law, which has since become the law of England and the Colonies, besides that of many foreign States, was laid down in the year 1602. It was some years, however, before popular feeling was satisfied with the action taken by King James II. One circumstance, however, which helped to set the public mind at rest was the publication of the King's Book of Bounty, a volume remarkable for its career, and which is reprinted in facsimile as a part of this work in Appendix I.

In 1621 the grievance had again assumed serious proportions, and an Act was passed actually comprising a declaration the King had made some years previously in a moment of candour. The fourth section of this Act was considered of great importance in protecting the public against monopolies; nothing is omitted which could make proceedings easy for an aggrieved person in assertion of his rights; in fact, it might be considered as the utmost Parliament could do to protect the weak against the strong. The growing tendency in our own times of patentees to threaten legal proceedings against all and sundry, has given patents a new and formidable character. The interests of the public as against that of the patentee is a matter of great and growing importance, and in which vast interests are involved.

The power to seize infringing goods is no longer included in a patent grant, but it is at the present day a common form of pleading to ask for the giving up of such goods, as part of the damages asked for, and the Courts do not shrink from allowing this in certain cases: it is a recent innovation, dating from the year 1846, and the author considers that it arises from what are, it is contended, insufficient authorities. The earliest report in which the author has been able to find an order for destruction is in 1864, but there seems to have been some confusion in its drawing up. But in *Tangye v. Stott*, in 1866, the defendant had the option of giving up or destroying the incriminating goods, so that it does not appear to be definitely recognised that the property in them may pass to the patentee.

The celebrated Case of Monopolies, which the author considers to be one of the most interesting reports in the English Law Books, is here, and now for the first time collected and published in complete form. Considered altogether, the author thinks "it is not too much to say that in the whole of legal history there is no other deliverance in any tongue which has proved to be so fruitful of results, nor any which has contributed more to the advancement of society in modern times."

The book cannot fail to be of great interest to those whose minds are cast in a legal mould, and it will undoubtedly be of great service to those whose business comes within its scope. Besides an excellent general Index, there are 19 pages of Cases which are referred to in the work, also carefully indexed.

CORRESPONDENCE.

RELATIONS BETWEEN MELTING-POINTS AND LATENT HEATS OF FUSION.

To the Editor of the Chemical News.

SIR,—In the CHEMICAL NEWS of June 11 (vol. lxxv., p. 278) there appeared a paper by Dr. Joseph W. Richards dealing with relations existing between the absolute melting-points (T) and the latent heats of fusion (L) of the metals. Dr. Richards, in 1893, announced that the latent heats of the metals bears a simple ratio to the heat required to raise the metal from the absolute zero to its melting-point.

In June, 1895, in a short paper I forwarded to the Chemical Society, I proposed this relation, which is practically the same as that of Dr. Richards:—"That where the valency is the same, the absolute temperature, L , of the melting-point is proportional to the quantity $\frac{L}{S}$; L being the latent heat of fusion, and S being the specific heat which has to be treated as a constant. The quantity $\frac{L}{S}$ I termed the temperature equivalent of fusion. Expressed in this way the numerical agreement was very striking.

Dr. Richards does not include the non-metals in expressing his relation; but between bromine and iodine there is an exact agreement, although the number obtained from these elements does not tally with that given by sodium, potassium, and silver—metallic monovalent elements—but is almost exactly the number given by the latter.

This relation of mine was proposed as a corollary to a relation brought forward by Mr. Holland Crompton before the Chemical Society, in April or May, 1895, when he proposed the relation $\frac{L}{AT} = KV$; A being the atomic weight, V the valency, and K a constant. Previous to this (in May or June, 1895), I had read a

paper before the Physical Society, in which I brought forward this relation,—

$$\left(T + \frac{L}{S} + \right)a = \text{constant},$$

as holding between the members of any family in the periodic classification of the elements; a being the mean coefficient of expansion between T and -273° . The values thus found were satisfactory, but the argument following which the relation was obtained was received with considerable adverse comment. This relation, combined with that of Mr. Holland Crompton, mentioned above, leads to these relations, with the same restriction as before:—

$$Ta = \text{constant}.$$

$$\frac{L}{S}a = \text{constant}.$$

The calculated values of these quantities were confirmation of the truth of the relations.

At that time I had not seen Pictet's rule,—

$$Ta\sqrt[3]{V} = \text{constant};$$

V being the atomic volume of the element in question. Now, if $Ta = \text{constant}$ for the members of a periodic family, and also $Ta\sqrt[3]{V} = \text{constant}$ generally, then must $\sqrt[3]{V}$ be constant for the members of a periodic family.

Dr. Richards concludes his paper by showing that, by combining his relation with Pictet's rule, the relation

$$L = 2.1 T = \frac{9.5}{a\sqrt[3]{V}} \text{ is obtained. On exactly the same}$$

grounds, by combining my relation $Ta = \text{constant}$ with Pictet's rule, we obtain the relation $L = 2.1 T = \frac{\text{constant}}{a}$.

This relation, as before, being restricted to members of the same periodic family.

Dr. Richards, in his paper, concludes with calculating certain unknown latent heats. For one of these (thallium) he gives the value 5.8; this has been determined by Messrs. Neville and Heycock—unless my memory misleads me—to be 5.1.

Dr. Richards quotes the latent heat of copper as 43; this quantity was not available when I read my paper before the Physical Society; I stated then that a value of about 40 would bring that element into line with its fellow members in its periodic relationships.

I regret that I am unable to quote any actual figures, but placed as I am—on a sugar estate some distance from a town—no data are available for me.

A short abstract of my paper appeared in the *CHEMICAL NEWS* (vol. lxxi., p. 303). The Physical Society did not think fit to publish it in their *Proceedings*.

In conclusion, I wish to say that what I have written is in a friendly spirit, and that I have no desire to break a lance with so redoubtable an antagonist as Dr. Richards.—I am, &c.,

NOËL DEERR.

The Laboratory,
Plantation, Windsor Forest,
West Coast, Demerara.

On Cafetannic Acid.—P. Cazeneuve and M. Haddon.—Recent researches based on the action of phenylhydrazine, either on cafetannic itself or on the sugar formed by its splitting up, show us that the formula adopted for cafetannic acid is entirely wrong, and that the sugar formed is a new sugar, which cannot be confounded with mannitan or with any known sugar.—*Journal de Pharmacie et Chemie*, vi., No. 2.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Journal de Pharmacie et Chemie.
Series 6, vol. v., No. 12.

Action of Sodium on Albumenoid Matters.—E. Lépineis.—The halogen proteids of Blum and the iodised albumen of Renault are very complex and variable in their composition and their action on organisms. The author has therefore directed his endeavours to producing a compound always identical and whose composition is well known: for this purpose he used an aqueous solution of iodine, and let it act in the cold, so as to avoid violent reactions. He has worked on casein as well as albumen. The experiments on pure casein were unsatisfactory owing to its feeble solubility, so he had recourse to milk itself, and to this he added a solution of iodised iodide (*iodé ioduré*) until a slight excess could be detected by means of chloroform, even after standing twenty-four hours. At the end of this time an equal volume of distilled water, containing a little acetic acid, is added; there is thus obtained a yellowish brown coagulum, which is well washed with water and treated with weak soda: the colour disappears, and a soluble caseinate is formed. From the filtered liquid we can again precipitate the casein by acetic acid. To obtain it perfectly pure this must be repeated five or six times; it then has a constant composition and leaves no ash on calcination. There is no doubt that this body contains iodine in its molecule, for if treated with soda and nitrate of soda, and taking up the mass with water, it is easy to detect the presence of iodine. We may therefore conclude that this casein is actually iodised. To show its constant composition five different analyses were made, and they gave average results of 21.6 per cent iodine and 14.15 per cent nitrogen, the greatest variations being 20.0 and 22.4 per cent for iodine and 14.1 and 14.23 per cent for nitrogen. The first yellowish brown coagulum loses its colour not only when treated with soda, but also by losing a portion of its iodine when treated with hyposulphite of soda or sulphurous acid; but in neither case is all the iodine removed. The author hopes that, owing to its constant proportion of iodine, this iodo-casein may become useful in therapeutics, and he proposes carrying out some experiments on digestion with men and animals.

Presence of Lead in some Sterilised Artificial Serums.—M. Chevreton.—A case of poisoning by an injection of an artificial serum having been observed, M. Chevreton sought for the cause, and he found that, by keeping an artificial physiological serum (7 per 1000 solution of NaCl) at 120° for twenty minutes, in different flasks, when lead glass was used the liquid became charged with this poison; and if this serum be used for subcutaneous or intra-venous injections, there is naturally a very grave risk of lead-poisoning: the presence of lead is easily proved by means of iodide of potassium.

Series 6, vol. vi., No. 1.

On a Method of Oxidation and Chlorination.—A. Villiers.—The addition of a trace of a salt of manganese will, in many cases, accelerate oxidation. This action can be explained by the production of easily decomposable salts, similar to those produced in the preparation of chlorine; the presence of traces of manganese can not only keep the oxidation in media containing special oxidising materials, but can in certain cases facilitate the direct absorption of oxygen from the air. The conditions under which oxidation takes place, under the influence of salts of manganese, in the case of oxalic acid, are rather curious. The disengagement of gas can be seen in the

cold. It will last several weeks, probably even several years. These reactions, of which several are already known, have the characteristics of fermentations produced by chemical ferments, and may be rightly classed as *mineral ferments*.

On Isomerism of Pilocarpidine and Pilocarpine.—A. Petit and M. Polonovski. — Two experiments have sufficed to satisfy the authors that the transformation of pilocarpine into pilocarpidine takes place inter-molecularly. After boiling a salt of pilocarpine with an excess of dilute soda for some hours, in a flask fitted with a vertical condenser, the condenser was lowered and three-quarters of the solution distilled, although nearly all the pilocarpine was transformed, they were unable to detect the least trace of methylic alcohol; *there is therefore no elimination of a methyl group* during the transformation of pilocarpine into pilocarpidine. Another experiment was still more decisive. Chlorhydrate of pilocarpine, kept in a state of fusion for a few instants, was entirely converted into chlorhydrate of pilocarpidine. Full details, weights, and temperatures, employed in this experiment, are given.

Contribution to the Study of the Preparation of Ordinary Ether.—L. Prunier.—In making ether from sulphuric acid and alcohol we are in the habit of regarding the transformation as in two successive phases, forming a complete cycle, being indefinitely renewed; this continuity does not exist in practice. The reaction certainly does take place in an acid medium, but the presence of sulphuric acid is not indispensable; etherification takes place in its absence, the acidity being due to sulphurous acid, sulphovinic acid, or its derivatives, the latter perhaps being of considerable importance. Isoethionic acid and its derivatives are taken as a type of the group, as their properties are well known, and in the author's experience the sulphonic derivatives have been characterised by groups and not by distinct species. Instead of admitting the continual regeneration of free sulphuric acid, the author prefers to think that the alcohol, added gradually, acts principally on the two sulphuric ethers, and above all on their products of decomposition,—the sulphonic derivatives, acid and neutral,—which constitute, in great part, the residues, and allow of the explanation of the phenomena observed.

Series 6, vol. vi., No. 2.

Research on the Rapid Estimation of Boric Acid (Preservative) in Milk.—G. Denigès.—The author expresses surprise at Mr. Farrington's recent results on the acidity of boric acid, and he thinks, after having repeated the experiments, that the latter has not obtained the correct results, though the *sense* of his experiments may be correct. By the modified method the author proposes, he claims that his results are exact when the quantity of boric acid present does not exceed 3 grms. per litre, and when the quantity of lactose present in the milk is from 40 to 50 grms. per litre. When these figures are exceeded it is necessary to dilute the milk.

Estimation of Glycerin by Bichromate of Potassium and Sulphuric Acid.—F. Bordas and Sig. de Raczkowski.—Already inserted.

Destruction of Organic Matter in Toxicology.—A. Villiers.—A very convenient mineral ferment, for the destruction of organic matter in toxicological research, can be produced by the aid of salt of manganese. The material to be treated is placed in a flask with dilute hydrochloric acid (3 to 1). Add a few drops of a solution of a manganese salt, and a little nitric acid, which must be renewed as it becomes used up. The mixture must be gently heated. The gases produced are carbonic acid and nearly pure nitrogen, and no disagreeable odours are evolved. Materials such as liver, lungs, &c., are dissolved in a few minutes; muscular fibre takes about an hour, and a fatty mass remains which resists the oxidising action of the mixture, and which appears to contain products of substitution.

On Pseudo-intestinal Calculus.—Marcel Delépine.—The author found, after considerable trouble and groping in the dark, that certain balls which had been sent to him to examine as intestinal calculi were merely undigested balls of bread crumb, which the patient had been in the habit of making and swallowing.

Revue Universelle des Mines et de la Metallurgie.
Series 3, vol. xxxviii., No. 3.

This number contains no matter of chemical interest.

Series 3, vol. xxxix., No. 1.

Saline Deposits of the Plains of Northern Germany.—Franz and Büttgenbach. — There are three rivers in Germany named *Saale*. This name is evidently derived from the numerous saline sources which are to be found in their valleys. In 1837 attempts were made to augment the supply of mineral waters by sinking shafts, but the work was abandoned, as it was found that the waters thus obtained "artificially" were not equal dietetically to those which rose naturally. But in 1861 the commercial value of the potassic salts (*kalisalze*) was recognised, and the industry at Stassfurt soon assumed colossal dimensions. The deposits are of a very complicated nature, and the deposition and formation of the beds must have proceeded and been interrupted many times and at different temperatures. There are at the present time twenty-five different species of deposits known; these are all given in the order of deposition. The most important of these salts are *sydnone*, KCl, and *kainite*, $K_2SO_4 \cdot MgSO_4 \cdot MgCl_2 \cdot 6H_2O$. These two salts are found in the ashes of most plants, and are for this reason much sought after as manures. The mean thickness of the beds of the potassic salts is at least 20 metres, and, considering the area covered, the author estimates the quantity available at 10 milliard (10,000,000,000) tons, and, at the present output of 3,000,000 tons a year, the beds will last for 33 centuries.

MISCELLANEOUS.

Obituary.—Professor Victor Meyer died a few days ago. His death seems to have been sudden; but according and trustworthy accounts of his end seem as yet wanting.

Sixty-ninth Meeting of the German Association of Natural Science and Medicine.—This Association will meet at Brunswick, after an absence of fifty-six years, on the 20th to the 25th of September, 1897. There will be two principal Sections, the first dealing with Natural Science; it is subdivided into three Sub-sections.

1. Section for Mathematics, Physics, Chemistry, Agriculture, Instruments, &c.
2. Section for Mineralogy, Botany, Zoology, Geography, &c.
3. Section for instruction in Mathematical and Natural Science.

The second principal Section is devoted to Medical Science, and comprises five Sub-sections.

1. Section for General Medicine: Pathology, Anatomy, Surgery, &c.
2. Section for Specialism: Nervous diseases, Ophthalmology, Laryngology, Dermatology, &c.
3. Section for Anatomical Physiology: Physiological Chemistry, Pharmacology.
4. Section for General Hygiene, Bacteriology, Climatology, Military Sanitation, Veterinary Science, &c.
5. Section for Pharmacy.

A large number of papers have been promised; these, after reading, will be followed by discussions. Though the sections will be all over on the 25th, the meeting will not break up till the 27th, the two extra days being devoted to excursions and social pleasure.

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DAVID LEWIS, Treasurer
Treasurer's Chambers, 20, York Place, Edinburgh, July 14th, 1897.

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F. T. STEAVENSON, Town Clerk.
Houndgate, Darlington, August 6, 1897.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1969.

BRITISH ASSOCIATION FOR THE ADVANCEMENT OF SCIENCE.

TORONTO, 1897.

INAUGURAL ADDRESS OF THE PRESIDENT,

SIR JOHN EVANS, K.C.B., D.C.L., LL.D., Sc.D.,
TREAS. R.S., V.P.S.A., FOR. SEC. G.S.,
CORRESPONDANT DE L'INSTITUT DE FRANCE, &c.

ONCE more has the Dominion of Canada invited the British Association for the Advancement of Science to hold one of the annual meetings of its members within the Canadian territory; and for a second time has the Association had the honour and pleasure of accepting the proffered hospitality.

In doing so the Association has felt that, if by any possibility the scientific welfare of a locality is promoted by its being the scene of such a meeting, the claims should be fully recognised of those who, though not dwelling in the British Isles, are still inhabitants of that Greater Britain whose prosperity is so intimately connected with the fortunes of the Mother Country.

Here, especially, as loyal subjects of one beloved Sovereign, the sixtieth year of whose beneficent reign has just been celebrated with equal rejoicing in all parts of her Empire; as speaking the same tongue, and as in most instances connected by the ties of one common parentage, we are bound together in all that can promote our common interests.

There is, in all probability, nothing that will tend more to advance those interests than the diffusion of science in all parts of the British Empire, and it is towards this end that the aspirations of the British Association are ever directed, even if in many instances the aim may not be attained.

We are, as already mentioned, indebted to Canada for previous hospitality; but we must also remember that, since the time when we last assembled on this side of the Atlantic, the Dominion has provided the Association with a President, Sir William Dawson, whose name is alike well known in Britain and America, and whose reputation is indeed world-wide. We rejoice that we have still among us the pioneer of American geology, who among other discoveries first made us acquainted with the "Air-breathers of the Coal," the terrestrial, or more properly arboreal, Saurians of the New Brunswick and Nova Scotia Coal-measures.

On our last visit to Canada, in 1884, our place of assembly was Montreal, a city which is justly proud of her McGill University; to-day we meet within the buildings of another of the Universities of this vast Dominion,—and in a city, the absolute fitness of which for such a purpose must have been foreseen by the native Indian tribes when they gave to a small aggregation of huts upon this spot the name of Toronto—"the place of meetings."

Our gathering this year presents a feature of entire novelty and extreme interest, inasmuch as the sister Association of the United States of America,—still mourning the loss of their illustrious President, Professor Cope,—and some other learned societies, have made special arrangements to allow of their members coming here to join us. I need hardly say how welcome their presence is, nor how gladly we look forward to their taking part in

our discussions, and aiding us by interchange of thought. To such a meeting the term "international" seems almost misapplied. It may rather be described as a family gathering, in which our relatives more or less distant in blood, but still intimately connected with us by language, literature, and habits of thought, have spontaneously arranged to take part.

The domain of Science is no doubt one in which the various nations of the civilised world meet upon equal terms, and for which no other passport is required than some evidence of having striven towards the advancement of natural knowledge. Here, on the frontier between the two great English-speaking nations of the world, who is there that does not inwardly feel that anything which conduces to an intimacy between the representatives of two countries, both of them actively engaged in the pursuit of science, may also, through such an intimacy, react on the affairs of daily life, and aid in preserving those cordial relations that have now for so many years existed between the great American Republic and the British Islands, with which her early foundations are indissolubly connected? The present year has witnessed an interchange of courtesies which has excited the warmest feelings of approbation on both sides of the Atlantic. I mean the return to its proper custodians of one of the most interesting of the relics of the Pilgrim Fathers, the Log of the "Mayflower." May this return, trifling in itself, be of happy augury as testifying to the feelings of mutual regard and esteem which animate the hearts both of the donors and of the recipients!

At our meeting in Montreal the President was an investigator who had already attained to a foremost place in the domains of Physics and Mathematics, Lord Rayleigh. In his address he dealt mainly with topics such as Light, Heat, Sound, and Electricity, on which he is one of our principal authorities. His name and that of his fellow-worker, Professor Ramsay, are now and will in all future ages be associated with the discovery of the new element, Argon. Of the ingenious methods by which that discovery was made, and the existence of Argon established, this is not the place to speak. One can only hope that the element will not always continue to justify its name by its inertness.

The claims of such a leader in physical science as Lord Rayleigh to occupy the Presidential chair are self-evident, but possibly those of his successor on this side of the Atlantic are not so immediately apparent. I cannot for a moment pretend to place myself on the same purely scientific level as my distinguished friend and for many years colleague, Lord Rayleigh, and my claims, such as they are, seem to me to rest on entirely different grounds.

Whatever little I may have indirectly been able to do in assisting to promote the advancement of science, my principal efforts have now for many years been directed towards attempting to forge those links in the history of the world, and especially of humanity, that connect the past with the present, and towards tracing that course of evolution which plays as important a part in the physical and moral development of man as it does in that of the animal and vegetable creation.

It appears to me, therefore, that my election to this important post may, in the main, be regarded as a recognition by this Association of the value of Archaeology as a science.

Leaving all personal considerations out of question, I gladly hail this recognition, which is, indeed, in full accordance with the attitude already for many years adopted by the Association towards Anthropology, one of the most important branches of true Archaeology.

It is no doubt hard to define the exact limits which are to be assigned to Archaeology as a science, and Archaeology as a branch of History and Belles Lettres. A distinction is frequently drawn between science on the one hand, and knowledge or learning on the other; but translate the terms into Latin, and the distinction at once disappears,

In illustration of this I need only cite Bacon's great work on the "Advancement of Learning," which was, with his own aid, translated into Latin under the title "*De Augmentis Scientiarum*."

It must, however, be acknowledged that a distinction does exist between Archæology proper and what—for want of a better word—may be termed Antiquarianism. It may be interesting to know the internal arrangements of a Dominican convent in the Middle Ages; to distinguish between the different mouldings characteristic of the principal styles of Gothic architecture; to determine whether an English coin bearing the name of Henry was struck under Henry II., Richard, John, or Henry III., or to decide whether some given edifice was erected in Roman, Saxon, or Norman times. But the power to do this, though involving no small degree of detailed knowledge and some acquaintance with scientific methods, can hardly entitle its possessors to be enrolled among the votaries of science.

A familiarity with all the details of Greek and Roman mythology and culture must be regarded as a literary rather than a scientific qualification; and yet when among the records of classical times we come upon traces of manners and customs which have survived for generations, and which seem to throw some rays of light upon the dim past, when history and writing were unknown, we are, I think, approaching the boundaries of scientific Archæology.

Every reader of Virgil knows that the Greeks were not merely orators, but that with a pair of compasses they could describe the movements of the heavens and fix the rising of the stars; but when by modern Astronomy we can determine the heliacal rising of some well-known star, with which the worship in some given ancient temple is known to have been connected, and can fix its position on the horizon at some particular spot, say, three thousand years ago, and then find that the axis of the temple is directed exactly towards that spot, we have some trustworthy scientific evidence that the temple in question must have been erected at a date approximately 1100 years B.C. If on or close to the same site we find that more than one temple was erected, each having a different orientation, these variations, following as they may fairly be presumed to do the changing position of the rising of the dominant star, will also afford a guide as to the chronological order of the different foundations. The researches of Mr. Penrose seem to show that in certain Greek temples, of which the date of foundation is known from history, the actual orientation corresponds with that theoretically deduced from astronomical data.

Sir J. Norman Lockyer has shown that what holds good for Greek temples applies to many of far earlier date in Egypt, though up to the present time hardly a sufficient number of accurate observations have been made to justify us in foreseeing all the instructive results that may be expected to arise from Astronomy coming to the aid of Archæology.

The intimate connection of Archæology with other sciences is in no case so evident as with respect to Geology, for, when considering subjects such as those I shall presently discuss, it is almost impossible to say where the one science ends and the other begins.

By the application of geological methods many archæological questions relating even to subjects on the borders of the historical period have been satisfactorily solved. A careful examination of the limits of the area over which its smaller coins are found has led to the position of many an ancient Greek city being accurately ascertained; while in England it has only been by treating the coins of the Ancient Britons, belonging to a period before the Roman occupation, as if they were actual fossils, that the territories under the dominion of the various kings and princes who struck them have been approximately determined. In arranging the chronological sequence of these coins, the evolution of their types—a process almost as remarkable, and certainly as

well-defined, as any to be found in Nature—has served as an efficient guide. I may venture to add that the results obtained from the study of the morphology of this series of coins were published ten years before the appearance of Darwin's great work on the "Origin of Species."

When we come to the consideration of the relics of the Early Iron and Bronze Ages, the aid of Chemistry has of necessity to be invoked. By its means we are able to determine whether the iron of a tool or weapon is of meteoritic or volcanic origin, or has been reduced from iron-ore, in which case considerable knowledge of metallurgy would be involved on the part of those who made it. With bronze antiquities the nature and extent of the alloys combined with the copper may throw light not only on their chronological position, but on the sources whence the copper, tin, and other metals of which they consist were originally derived. I am not aware of there being sufficient differences in the analyses of the native copper from different localities in the region in which we are assembled, for Canadian Archæologists to fix the sources from which the metal was obtained which was used in the manufacture of the ancient tools and weapons of copper that are occasionally discovered in this part of the globe.

Like Chemistry, Mineralogy and Petrology may be called to the assistance of Archæology in determining the nature and source of the rocks of which ancient stone implements are made; and, thanks to researches of the followers of those sciences, the old view that all such implements formed of jade and found in Europe must of necessity have been fashioned from material imported from Asia can no longer be maintained. In one respect the Archæologist differs in opinion from the Mineralogist—namely, as to the propriety of chipping off fragments from perfect and highly finished specimens for the purpose of submitting them to microscopic examination.

I have hitherto been speaking of the aid that other sciences can afford to Archæology when dealing with questions that come almost, it not quite, within the fringe of history, and belong to times when the surface of our earth presented much the same configuration as regards the distribution of land and water, and hill and valley, as it does at present, and when, in all probability, the climate was much the same as it now is. When, however, we come to discuss that remote age in which we find the earliest that are at present known of Man's appearance upon earth, the aid of Geology and Palæontology becomes absolutely imperative.

The changes in the surface configuration and in the extent of the land, especially in a country like Britain, as well as the modifications of the fauna and flora since those days, have been such that the Archæologist pure and simple is incompetent to deal with them, and he must either himself undertake the study of these other sciences or call experts in them to his assistance. The evidence that Man had already appeared upon the earth is afforded by stone implements wrought by his hands, and it falls strictly within the province of the Archæologist to judge whether given specimens were so wrought or not; it rests with the Geologist to determine their stratigraphical or chronological position, while the Palæontologist can pronounce upon the age and character of the associated fauna and flora.

If left to himself the Archæologist seems too prone to build up theories founded upon form alone, irrespective of geological conditions. The Geologist, unaccustomed to archæological details, may readily fail to see the difference between the results of the operations of Nature and those of Art, and may be liable to trace the effects of man's handiwork in the chipping, bruising, and wearing which in all ages result from natural forces; but the united labours of the two, checked by those of the Palæontologist, cannot do otherwise than lead towards sound conclusions.

It will perhaps be expected of me that I should on the present occasion bring under review the state of our

present knowledge with regard to the Antiquity of Man; and probably no fitter place could be found for the discussion of such a topic than the adopted home of my venerated friend, the late Sir Daniel Wilson, who first introduced the word "pre-historic" into the English language.

Some among us may be able to call to mind the excitement, not only among men of science but among the general public, when, in 1859, the discoveries of M. Boucher de Perthes and Dr. Rigollot in the gravels of the valley of the Somme, at Abbeville and Amiens, were confirmed by the investigations of the late Sir Joseph Prestwich, myself, and others, and the co-existence of Man with the extinct animals of the Quaternary fauna, such as the mammoth and woolly-haired rhinoceros, was first virtually established. It was at the same time pointed out that these relics belonged to a far earlier date than the ordinary stone weapons found upon the surface, which usually showed signs of grinding or polishing, and that in fact there were two Stone Ages in Britain. To these the terms Neolithic and Palæolithic were subsequently applied by Sir John Lubbock.

The excitement was not less when, at the meeting of this Association at Aberdeen in the autumn of that year, Sir Charles Lyell, in the presence of the Prince Consort, called attention to the discoveries in the valley of the Somme, the site of which he had himself visited, and to the vast lapse of time indicated by the position of the implements in drift-deposits a hundred feet above the existing river.

The conclusions forced upon those who examined the facts on the spot did not receive immediate acceptance by all who were interested in Geology and Archæology, and fierce were the controversies on the subject that were carried on both in the newspapers and before various learned societies.

It is at the same time instructive and amusing to look back on the discussions of those days. While one class of objectors accounted for the configuration of the flint implements from the gravels by some unknown chemical agency, by the violent and continued gyratory action of water, by fracture resulting from pressure, by rapid cooling when hot or by rapid heating when cold, or even regarded them as aberrant forms of fossil fishes, there were others who, when compelled to acknowledge that the implements were the work of men's hands, attempted to impugn and set aside the evidence as to the circumstances under which they had been discovered. In doing this they adopted the view that the worked flints had either been introduced into the containing beds at a comparatively recent date, or if they actually formed constituent parts of the gravel then that this was a mere modern alluvium resulting from floods at no very remote period.

In the course of a few years the main stream of scientific thought left this controversy behind, though a tendency to cut down the lapse of time necessary for all the changes that have taken place in the configuration of the surface of the earth and in the character of its occupants since the time of the Palæolithic gravels, still survives in the inmost recesses of the hearts of not a few observers.

In his Address to this Association at the Bath meeting of 1864, Sir Charles Lyell struck so true a note that I am tempted to re-produce the paragraph to which I refer:—

"When speculations on the long series of events which occurred in the glacial and post-glacial periods are indulged in, the imagination is apt to take alarm at the immensity of the time required to interpret the monuments of these ages, all referable to the era of existing species. In order to abridge the number of centuries which would otherwise be indispensable, a disposition is shown by many to magnify the rate of change in pre-historic times by investing the causes which have modified the animate and inanimate world with extraordinary and excessive energy. It is related of a great Irish orator of our day that when he was about to contribute somewhat parsimoniously towards a public charity,

he was persuaded by a friend to make a more liberal donation. In doing so he apologised for his first apparent want of generosity by saying that his early life had been a constant struggle with scanty means, and that 'they who are born to affluence cannot easily imagine how long a time it takes to get the chill of poverty out of one's bones.' In like manner we of the living generation, when called upon to make grants of thousands of centuries in order to explain the events of what is called the modern period, shrink naturally at first from making what seems so lavish an expenditure of past time. Throughout our early education we have been accustomed to such strict economy in all that relates to the chronology of the earth and its inhabitants in remote ages, so fettered have we been by old traditional beliefs, that even when our reason is convinced, and we are persuaded that we ought to make more liberal grants of time to the Geologist, we feel how hard it is to get the chill of poverty out of our bones."

Many, however, have at the present day got over this feeling, and of late years the general tendency of those engaged upon the question of the antiquity of the human race has been in the direction of seeking for evidence by which the existence of Man upon the earth could be carried back to a date earlier than that of the Quaternary gravels.

There is little doubt that such evidence will be eventually forthcoming, but, judging from all probability, it is not in Northern Europe that the cradle of the human race will eventually be discovered, but in some part of the world more favoured by a tropical climate, where abundant means of subsistence could be procured, and where the necessity for warm clothing did not exist.

Before entering into speculations on this subject, or attempting to lay down the limits within which we may safely accept recent discoveries as firmly established, it will be well to glance at some of the cases in which implements are stated to have been found under circumstances which raise a presumption of the existence of Man in pre-Glacial, Pliocene, or even Miocene times.

Flint implements of ordinary Palæolithic type have, for instance, been recorded as found in the Eastern Counties of England, in beds beneath the Chalky Boulder Clay; but on careful examination the geological evidence has not to my mind proved satisfactory, nor has it, I believe, been generally accepted. Moreover, the archæological difficulty that Man, at two such remote epochs as the pre-Glacial and the post-Glacial, even if the term Glacial be limited to the Chalky Boulder Clay, should have manufactured implements so identical in character that they cannot be distinguished apart, seems to have been entirely ignored.

Within the last few months we have had the report of worked flints having been discovered in the late Pliocene Forest Bed of Norfolk; but in that instance the signs of human workmanship upon the flints are by no means apparent to all observers.

But such an antiquity as the Forest Bed is as nothing when compared with that which would be implied by the discoveries of the work of men's hands in the Pliocene and Miocene Beds of England, France, Italy, and Portugal, which have been accepted by some geologists. There is one feature in these cases which has hardly received due attention, and that is the isolated character of the reputed discoveries. Had man, for instance, been present in Britain during the Crag Period, it would be strange indeed if the sole traces of his existence that he left were a perforated tooth of a large shark, the sawn rib of a manatee, and a beaming full face, carved on the shell of a peccunculus!

In an address to the Anthropological Section at the Leeds meeting of this Association, in 1890, I dealt somewhat fully with these supposed discoveries of the remains of human art in beds of Tertiary date, and I need not here go further into the question. Suffice it to say that I see no reason why the verdict of "not proven" at which I then arrived should be reversed.

In the case of a more recent discovery in Upper Burma in beds at first pronounced to be Upper Miocene, but subsequently "definitely ascertained to be Pliocene," some of the flints are of purely natural and not artificial origin, so that two questions arise:—First, Were the fossil remains associated with the worked flints or with those of natural forms? And second, Were they actually found in the bed to which they have been assigned, or did they merely lie together on the surface?

Even the *Pithecanthropus erectus* of Dr. Eugene Dubois from Java meets with some incredulous objectors from both the physiological and the geological sides. From the point of view of the latter the difficulty lies in determining the exact age of what are apparently alluvial beds in the bottom of a river valley.

When we return to Palæolithic man, it is satisfactory to feel that we are treading on comparatively secure ground, and that the discoveries of the last forty years in Britain alone enable us to a great extent to re-constitute his history. We may not know the exact geological period when first he settled in the British area, but we have good evidence that he occupied it at a time when the configuration of the surface was entirely different from what it is at present: when the river valleys had not been cut down to anything like their existing depth, when the fauna of the country was of a totally different character from that of the present day, when the extension of the southern part of the island seaward was in places such that the land was continuous with that of the continent, and when in all probability a far more rainy climate prevailed. We have proofs of the occupation of the country by man during the long lapse of time that was necessary for the excavation of the river valleys. We have found the old floors on which his habitations were fixed, we have been able to trace him at work on the manufacture of flint instruments, and by building up the one upon the other the flakes struck off by the primæval workman in those remote times we have been able to reconstruct the blocks of flint which served as his material.

That the duration of the Palæolithic Period must have extended over an almost incredible length of time is sufficiently proved by the fact that valleys, some miles in width and of a depth of from 100 to 150 feet, have been eroded since the deposit of the earliest implement-bearing beds. Nor is the apparent duration of this period diminished by the consideration that the floods which hollowed out the valleys were not in all probability of such frequent occurrence as to teach Palæolithic man by experience the danger of settling too near to the streams, for had he kept to the higher slopes of the valley there would have been but little chance of his implements having so constantly formed constituent parts of the gravels deposited by the floods.

The examination of British cave-deposits affords corroborative evidence of this extended duration of the Palæolithic Period. In Kent's Cavern at Torquay, for instance, we find in the lowest deposit, the breccia below the red cave earth, implements of flint and chert corresponding in all respects with those of the high level and most ancient river gravels. In the cave-earth these are scarcer, though implements occur which also have their analogues in the river deposits; but, what is more remarkable, harpoons of reindeer's horn and needles of bone are present, identical in form and character with those of the caverns of the Reindeer Period in the South of France, and suggestive of some bond of union or identity of descent between the early troglodytes, whose habitations were geographically so widely separated the one from the other.

In a cavern at Creswell Crags, on the confines of Derbyshire and Nottinghamshire, a bone has moreover been found engraved with a representation of parts of a horse in precisely the same style as the engraved bones of the French caves.

It is uncertain whether any of the River-drift specimens belong to so late a date as these artistic cavern-remains;

but the greatly superior antiquity of even these to any Neolithic relics is testified by the thick layer of stalagmite, which had been deposited in Kent's Cavern before its occupation by men of the Neolithic and Bronze Periods.

Towards the close of the period covered by the human occupation of the French caves, there seems to have been a dwindling in the number of the larger animals constituting the Quaternary fauna, whereas their remains are present in abundance in the lower and therefore more recent of the valley gravels. This circumstance may afford an argument in favour of regarding the period represented by the later French caves as a continuation of that during which the old river gravels were deposited, and yet the great change in the fauna that has taken place since the latest of the cave-deposits included in the Palæolithic Period is indicative of an immense lapse of time.

How much greater must have been the time required for the more conspicuous change between the old Quaternary fauna of the river gravels and that characteristic of the Neolithic Period!

As has been pointed out by Prof. Boyd Dawkins, only thirty-one out of the forty-eight well-ascertained species living in the post-Glacial or River-drift Period survived into pre-historic or Neolithic times. We have not, indeed, any means at command for estimating the number of centuries which such an important change indicates; but when we remember that the date of the commencement of the Neolithic or Surface Stone Period is still shrouded in the mist of a dim antiquity, and that prior to that commencement the River-drift period had long come to an end; and when we further take into account the almost inconceivable ages that even under the most favourable conditions the excavation of wide and deep valleys by river action implies, the remoteness of the date at which the Palæolithic Period had its beginning almost transcends our powers of imagination.

We find distinct traces of river action from 100 to 200 feet above the level of existing streams and rivers, and sometimes at a great distance from them; we observe old fresh-water deposits on the slopes of valleys several miles in width; we find that long and lofty escarpments of rock have receded unknown distances since their summits were first occupied by Palæolithic man; we see that the whole side of a wide river valley has been carried away by invasion of the sea, which attacked and removed a barrier of chalk cliffs from 400 to 600 feet in height; we find that what was formerly an inland river has been widened out into an arm of the sea, now the highway of our fleets, and that gravels which were originally deposited in the bed of some ancient river now cap isolated and lofty hills.

And yet, remote as the date of the first known occupation of Britain by man may be, it belongs to what, geologically speaking, must be regarded as a quite recent period, for we are now in a position to fix with some degree of accuracy its place on the geological scale. Thanks to investigations ably carried out at Hoxne in Suffolk, and at Hitchin in Hertfordshire, by Mr. Clement Reid, under the auspices of this Association and of the Royal Society, we know that the implement-bearing beds at those places undoubtedly belong to a time subsequent to the deposit of the Great Chalky Boulder Clay of the Eastern Counties of England. It is, of course, self-evident that this vast deposit, in whatever manner it may have been formed, could not, for centuries after its deposition was complete, have presented a surface inhabitable by man. Moreover, at a distance but little farther north, beds exist which also—though at a somewhat later date—were apparently formed under Glacial conditions. At Hoxne the interval between the deposit of the Boulder Clay and of the implement-bearing beds is distinctly proved to have witnessed at least two noteworthy changes in climate. The beds immediately reposing on the Clay are characterised by the presence of alder in abundance, of hazel, and yew, as well as by that of numerous flower-

ing plants indicative of a temperate climate very different from that under which the Boulder Clay itself was formed. Above these beds characterised by temperate plants, comes a thick and more recent series of strata, in which leaves of the dwarf Arctic willow and birch abound, and which were in all probability deposited under conditions like those of the cold regions of Siberia and North America.

At a higher level and of more recent date than these—from which they are entirely distinct—are the beds containing Palæolithic implements, formed in all probability under conditions not essentially different from those of the present day. However this may be, we have now conclusive evidence that the Palæolithic implements are, in the Eastern Counties of England, of a date long posterior to that of the Great Chalky Boulder Clay.

It may be said, and said truly, that the implements at Hoxne cannot be shown to belong to the beginning rather than to some later stage of the Palæolithic Period.

The changes, however, that have taken place at Hoxne in the surface configuration of the country prove that the beds containing the implements cannot belong to the close of that period.

It must, moreover, be remembered that in what are probably the earliest of the Palæolithic deposits of the Eastern Counties, those at the highest level, near Brandon in Norfolk, where the gravels contain the largest proportion of pebbles derived from Glacial beds, some of the implements themselves have been manufactured from materials not native to the spot, but brought from a distance, and derived in all probability either from the Boulder Clay or from some of the beds associated with it.

We must, however, take a wider view of the whole question, for it must not for a moment be supposed that there are the slightest grounds for believing that the civilisation, such as it was, of the Palæolithic Period originated in the British Isles. We find in other countries implements so identical in form and character with British specimens that they might have been manufactured by the same hands. These occur over large areas in France under similar conditions to those that prevail in England. The same forms have been discovered in the ancient river gravels of Italy, Spain, and Portugal. Some few have been recorded from the north of Africa, and analogous types occur in considerable numbers in the south of that continent. On the banks of the Nile, many hundreds of feet above its present level, implements of the European types have been discovered; while in Somaliland, in an ancient river valley at a great elevation above the sea, Mr. Seton-Karr has collected a large number of implements formed of flint and quartzite, which, judging from their form and character, might have been dug out of the drift deposits of the Somme or the Seine, the Thames or the ancient Solent.

In the valley of the Euphrates implements of the same kind have also been found, and again farther east in the lateritic deposits of Southern India they have been obtained in considerable numbers. It is not a little remarkable, and it is at the same time highly suggestive, that a form of implement almost peculiar to Madras re-appears among implements from the very ancient gravels of the Manzanares at Madrid. In the case of the African discoveries we have as yet no definite Palæontological evidence by which to fix their antiquity; but in the Narbadâ Valley of Western India Palæolithic implements of quartzite seem to be associated with a local fauna of Pleistocene age, comprising, like that of Europe, the elephant, hippopotamus, ox, and other mammals of species now extinct. A correlation of the two faunas with a view of ascertaining their chronological relations is beset with many difficulties, but there seems reason for accepting this Indian Pleistocene fauna as in some degree more ancient than the European.

Is this not a case in which the imagination may be fairly invoked in aid of science? May we not from these data attempt in some degree to build up and reconstruct

the early history of the human family? There, in Eastern Asia, in a tropical climate, with the means of subsistence readily at hand, may we not picture to ourselves our earliest ancestors gradually developing from a lowly origin, acquiring a taste for hunting, if not indeed being driven to protect themselves from the beasts around them, and evolving the more complicated forms of tools or weapons from the simpler flakes which had previously served them as knives? May we not imagine that, when once the stage of civilisation denoted by these Palæolithic implements had been reached, the game for the hunter became scarcer, and that his life in consequence assumed a more nomad character? Then, and possibly not till then, may a series of migrations to "fresh woods and pastures new" not unnaturally have ensued, and these, following the usual course of "westward towards the setting sun," might eventually lead to a Palæolithic population finding its way to the extreme borders of Western Europe, where we find such numerous traces of its presence.

How long a term of years may be involved in such a migration it is impossible to say, but that such a migration took place the phenomena seem to justify us in believing. It can hardly be supposed that the process that I have shadowed forth was reversed, and that Man, having originated in North-Western Europe, in a cold climate where clothing was necessary and food scarce, subsequently migrated eastward to India and southward to the Cape of Good Hope! As yet, our records of discoveries in India and Eastern Asia are but scanty; but it is there that the traces of the cradle of the human race are, in my opinion, to be sought, and possibly future discoveries may place upon a more solid foundation the visionary structure that I have ventured to erect.

It may be thought that my hypothesis does not do justice to what Sir Thomas Browne has so happily termed "that great antiquity, America." I am, however, not here immediately concerned with the important Neolithic remains of all kinds with which this great continent abounds. I am now confining myself to the question of Palæolithic man and his origin, and in considering it I am not unmindful of the Trenton implements, though I must content myself by saying that the "turtleback" form is essentially different from the majority of those on the wide dissemination of which I have been speculating, and, moreover, as many here present are aware, the circumstances of the finding of these American implements are still under careful discussion.

Leaving them out of the question for the present, it may be thought worth while to carry our speculations rather further, and to consider the relations in time between the Palæolithic and the Neolithic Periods. We have seen that the stage in human civilisation denoted by the use of the ordinary forms of Palæolithic implements must have extended over a vast period of time if we have to allow for the migration of the primeval hunters from their original home, wherever it may have been in Asia or Africa, to the west of Europe, including Britain. We have seen that, during this migration, the forms of the weapons and tools made from silicious stones had become, as it were, stereotyped, and further, that, during the subsequent extended period implied by the erosion of the valleys, the modifications in the form of the implements and the changes in the fauna associated with the men who used them were but slight.

At the close of the period during which the valleys were being eroded comes that represented by the latest occupation of the caves by Palæolithic man, when both in Britain and in the South of France the reindeer was abundant; but among the stone weapons and implements of that long troglodytic phase of man's history not a single example with the edge sharpened by grinding has as yet been found. All that can safely be said is that the larger implements as well as the larger mammals had become scarcer, that greater power in chipping flint had been attained, that the arts of the engraver and the

sculptor had considerably developed, and that the use of the bow had probably been discovered.

Directly we encounter the relics of the Neolithic Period, often, in the case of the caves lately mentioned, separated from the earlier remains by a thick layer of underlying stalagmite, we find flint hatchets polished at the edge and on the surface, cutting at the broad and not at the narrow end, and other forms of implements associated with a fauna in all essential respects identical with that of the present day.

Were the makers of these polished weapons the direct descendants of Palæolithic ancestors whose occupation of the country was continuous from the days of the old river gravels? or had these long since died out, so that after Western Europe had for ages remained uninhabited, it was re-peopled in Neolithic times by the immigration of some new race of men? Was there, in fact, a "great gulf fixed" between the two occupations? or was there in Europe a gradual transition from the one stage of culture to the other?

It has been said that "what song the Syrens sang, or what name Achilles assumed when he hid himself among women, though puzzling questions, are not beyond all conjecture"; and though the questions now proposed may come under the same category, and must await the discovery of many more essential facts before they receive definite and satisfactory answers, we may, I think, throw some light upon them if we venture to take a few steps upon the seductive if insecure paths of conjecture. So far as I know we have as yet no trustworthy evidence of any transition from the one age to the other, and the gulf between them remains practically unbridged. We can, indeed, hardly name the part of the world in which to seek for the cradle of Neolithic civilisation, though we know that traces of what appear to have been a stone-using people have been discovered in Egypt, and that what must be among the latest of the relics of their industry have been assigned to a date some 3500 to 4000 years before our era. The men of that time had attained to the highest degree of skill in working flint that has ever been reached. Their beautifully made knives and spear-heads seem indicative of a culminating point reached after long ages of experience; but whence these artists in flint came or who they were is at present absolutely unknown, and their handiworks afford no clue to help us in tracing their origin.

Taking a wider survey, we may say that, generally speaking, not only the fauna but the surface configuration of the country were, in Western Europe at all events, much the same at the commencement of the Neolithic Period as they are at the present day. We have, too, no geological indications to aid us in forming any chronological scale.

The occupation of some of the caves in the south of France seems to have been carried on after the erosion of the neighbouring river valleys had ceased, and so far as our knowledge goes these caves offer evidence of being the latest in time of those occupied by Man during the Palæolithic Period. It seems barely possible that, though in the north of Europe there are no distinct signs of such late occupation, yet that, in the south, Man may have lived on, though in diminished numbers; and that in some of the caves, such, for instance, as those in the neighbourhood of Mentone, there may be traces of his existence during the transitional period that connects the Palæolithic and Neolithic Ages. If this were really the case, we might expect to find some traces of a dissemination of Neolithic culture from a North Italian centre, but I much doubt whether any such traces actually exist.

If it had been in that part of the world that the transition took place, how are we to account for the abundance of polished stone hatchets found in Central India? Did Neolithic man return eastward by the same route as that by which in remote ages his Palæolithic predecessor had migrated westward? Would it not be in defiance of all probability to answer such a question in the affirmative?

We have, it must be confessed, nothing of a substantial character to guide us in these speculations; but, pending the advent of evidence to the contrary, we may, I think, provisionally adopt the view that owing to failure of food, climatal changes, or other causes, the occupation of Western Europe by Palæolithic man absolutely ceased, and that it was not until after an interval of long duration that Europe was re-peopled by a race of men immigrating from some other part of the globe where the human race had survived, and in course of ages had developed a higher state of culture than that of Palæolithic man.

I have been carried away by the liberty allowed for conjecture into the regions of pure imagination, and must now return to the realms of fact, and one fact on which I desire for a short time to insist is that of the existence at the present day, in close juxtaposition with our own civilisation, of races of men who, at all events but a few generations ago, lived under much the same conditions as did our own Neolithic predecessors in Europe.

The manners and customs of these primitive tribes and peoples are changing day by day, their languages are becoming obsolete, their myths and traditions are dying out, their ancient processes of manufacture are falling into oblivion, and their numbers are rapidly diminishing, so that it seems inevitable that ere long many of these interesting populations will become absolutely extinct. The admirable Bureau of Ethnology instituted by our neighbours in the United States of America has done much towards preserving a knowledge of the various native races in this vast continent; and here in Canada the annual Archæological Reports presented to the Minister of Education are rendering good service in the same cause.

Moreover, the Committee of this Association appointed to investigate the physical characters, languages, and industrial and social conditions of the North-Western tribes of the Dominion of Canada is about to present its twelfth and final report, which in conjunction with those already presented will do much towards preserving a knowledge of the habits and languages of those tribes. It is sad to think that Mr. Horatio Hale, whose comprehensive grasp of the bearings of ethnological questions, and whose unremitting labours have so materially conduced to the success of the Committee, should be no longer among us. Although this report is said to be final, it is to be hoped that the Committee may be able to indicate lines upon which future work in the direction of ethnological and archæological research may be profitably carried on in this part of Her Majesty's dominions.

It is, however, lamentable to notice how little is being or has been officially done towards preserving a full record of the habits, belief, arts, myths, languages, and physical characteristics of the countless other tribes and nations more or less uncivilised which are comprised within the limits of the British Empire. At the meeting of this Association held last year at Liverpool it was resolved by the General Committee "that it is of urgent importance to press upon the Government the necessity of establishing a Bureau of Ethnology for Greater Britain, which by collecting information with regard to the native races within and on the borders of the Empire will prove of immense value to science and to the Government itself." It has been suggested that such a bureau might with the greatest advantage and with the least outlay and permanent expense be connected either with the British Museum or with the Imperial Institute, and the project has already been submitted for the consideration of the Trustees of the former establishment.

The existence of an almost unrivalled ethnological collection in the museum, and the presence there of officers already well versed in ethnological research, seem to afford an argument in favour of the proposed bureau being connected with it. On the other hand, the Imperial Institute was founded with an especial view to its being a centre around which every interest connected with the dependencies of the Empire might gather for information and support. The establishment within the last twelve

months of a Scientific Department within the Institute, with well-appointed laboratories and a highly trained staff, shows how ready are those concerned in its management to undertake any duties that may conduce to the welfare of the outlying parts of the British Empire; a fact of which I believe that Canada is fully aware. The Institute is therefore likely to develop, so far as its scientific department is concerned, into a Bureau of advice in all matters scientific and technical, and certainly a Bureau of Ethnology such as that suggested would not be out of place within its walls.

Wherever such an institution is to be established, the question of its existence must of necessity rest with Her Majesty's Government and Treasury, inasmuch as without funds, however moderate, the undertaking cannot be carried on. I trust that in considering the question it will always be borne in mind that in the relations between civilised and uncivilised nations and races it is of the first importance that the prejudices and especially the religious or semi-religious and caste prejudices of the latter should be thoroughly well known to the former. If but a single "little war" could be avoided in consequence of the knowledge acquired and stored up by the Bureau of Ethnology preventing such a misunderstanding as might culminate in warfare, the cost of such an institution would quickly be saved.

I fear that it will be thought that I have dwelt too long on primeval man and his modern representatives, and that I should have taken this opportunity to discuss some more general subject, such as the advances made in the various departments of science since last this Association met in Canada. Such a subject would no doubt have afforded an infinity of interesting topics on which to dilate. Spectrum analysis, the origin and nature of celestial bodies, photography, the connection between heat, light, and electricity, the practical applications of the latter, terrestrial magnetism, the liquefaction and solidification of gases, the behaviour of elements and compounds under the influence of extreme cold, the nature and uses of the Röntgen rays, the advances in bacteriology and in prophylactic medicine, might all have been passed under review, and to many of my audience would have seemed to possess greater claims to attention than the subject that I have chosen.

It must, however, be borne in mind that most, if not indeed all, of these topics will be discussed by more competent authorities in the various Sections of the Association by means of the Presidential addresses or otherwise. Nor must it be forgotten that I occupy this position as a representative of Archæology, and am therefore justified in bringing before you a subject in which every member of every race of mankind ought to be interested—the antiquity of the human family and the scenes of its infancy.

Others will direct our thoughts in other directions, but the farther we proceed the more clearly shall we realise the connection and interdependence of all departments of science. Year after year, as meetings of this Association take place, we may also foresee that "many shall run to and fro and knowledge shall be increased." Year after year advances will be made in science, and in reading that Book of Nature that lies ever open before our eyes; successive stones will be brought for building up that Temple of Knowledge of which our fathers and we have laboured to lay the foundations. May we not well exclaim with old Robert Recorde?—

"Oh worthy temple of Goddes magnificence: Oh throne of glorye and seate of the lorde: thy substance most pure what tonge can describe? thy signes are so wonderous, surmountinge mannes witte, the effects of thy motions so diuers in kinde: so harde for to seache, and worse for to fynde—Thy woorkes are all wonderous, thy cunning unknown: yet seedes of all knowledge in that booke are sown—And yet in that booke who rightly can reade, to all secrete knowledge it will him straighte leade" (Preface to Robert Recorde's *Castle of Knowledge*, 1556).

ADDRESS TO THE CHEMICAL SECTION OF THE BRITISH ASSOCIATION.

TORONTO, 1897.

By Professor WILLIAM RAMSAY, Ph.D., LL.D., Sc.D., F.R.S.,
President of the Section.

An Undiscovered Gas.

A SECTIONAL address to members of the British Association falls under one of three heads. It may be historical, or actual, or prophetic; it may refer to the past, the present, or the future. In many cases, indeed in all, this classification overlaps. Your former Presidents have given sometimes a historical introduction, followed by an account of the actual state of some branch of our science, and, though rarely, concluding with prophetic remarks. To those who have an affection for the past, the historical side appeals forcibly; to the practical man, and to the investigator engaged in research, the actual, perhaps, presents more charm, while to the general public, to whom novelty is often more of an attraction than truth, the prophetic aspect excites most interest. In this address I must endeavour to tickle all palates; and perhaps I may be excused if I take this opportunity of indulging in the dangerous luxury of prophecy, a luxury which the managers of scientific journals do not often permit their readers to taste.

The subject of my remarks to-day is a new gas. I shall describe to you later its curious properties; but it would be unfair not to put you at once in possession of the knowledge of its most remarkable property—it has not yet been discovered. As it is still unborn, it has not yet been named. The naming of a new element is no easy matter. For there are only twenty-six letters in our alphabet, and there are already over seventy elements. To select a name expressible by a symbol which has not already been claimed by one of the known elements is difficult, and the difficulty is enhanced when it is at the same time required to select a name which shall be descriptive of the properties (or want of properties) of the element.

It is now my task to bring before you the evidence for the existence of this undiscovered element.

It was noticed by Döbereiner, as long ago as 1817, that certain elements could be arranged in groups of three. The choice of the elements selected to form these triads was made on account of their analogous properties, and on the sequence of their atomic weights, which had at that time only recently been discovered. Thus calcium, strontium, and barium formed such a group; their oxides, lime, strontia, and baryta are all easily slaked, combining with water to form soluble lime-water, strontia-water, and baryta-water. Their sulphates are all sparingly soluble, and resemblance had been noticed between their respective chlorides and between their nitrates. Regularity was also displayed by their atomic weights. The numbers then accepted were 20, 42.5, and 65; and the atomic weight of strontium, 42.5, is the arithmetical mean of those of the other two elements, for $(65+20)/2=42.5$. The existence of other similar groups of three was pointed out by Döbereiner, and such groups became known as "Döbereiner's triads."

Another method of classifying the elements, also depending on their atomic weights, was suggested by Pettenkofer, and afterwards elaborated by Kremers, Gladstone, and Cooke. It consisted in seeking for some expression which would represent the differences between the atomic weights of certain allied elements. Thus, the difference between the atomic weight of lithium, 7, and sodium, 23, is 16; and between that of sodium and of potassium, 39, is also 16. The regularity is not always so conspicuous; Dumas, in 1857, contrived a somewhat complicated expression which, to some extent, exhibited regularity in the atomic weights of fluorine, chlorine, bro-

mine, and iodine; and also of nitrogen, phosphorus, arsenic, antimony, and bismuth.

The upshot of these efforts to discover regularity was that, in 1864, Mr. John Newlands, having arranged the elements in eight groups, found that when placed in the order of their atomic weights, "the eighth element, starting from a given one, is a kind of repetition of the first, like the eighth note of an octave in music." To this regularity he gave the name "The Law of Octaves."

The development of this idea, as all chemists know, was due to the late Professor Lothar Meyer, of Tübingen, and to Professor Mendeléeff, of St. Petersburg. It is generally known as the "Periodic Law." One of the simplest methods of showing this arrangement is by means of a cylinder divided into eight segments by lines drawn parallel to its axis; a spiral line is then traced round the cylinder, which will, of course, be cut by these lines eight times at each revolution. Holding the cylinder vertically, the name and atomic weight of an element is written at each intersection of the spiral with a vertical line, following the numerical order of the atomic weights. It will be found, according to Lothar Meyer and Mendeléeff, that the elements grouped down each of the vertical lines form a natural class; they possess similar properties, form similar compounds, and exhibit a graded relationship between their densities, melting-points, and many of their other properties. One of these vertical columns, however, differs from the others, inasmuch as on it there are three groups, each consisting of three elements with approximately equal atomic weights. The elements in question are iron, cobalt, and nickel; palladium, rhodium, and ruthenium; and platinum, iridium, and osmium. There is apparently room for a fourth group of three elements in this column, and it may be a fifth. And the discovery of such a group is not unlikely, for when this table was first drawn up Professor Mendeléeff drew attention to certain gaps, which have since been filled up by the discovery of gallium, germanium, and others.

The discovery of argon at once raised the curiosity of Lord Rayleigh and myself as to its position in this table. With a density of nearly 20, if a diatomic gas, like oxygen and nitrogen, it would follow fluorine in the periodic table; and our first idea was that argon was probably a mixture of three gases, all of which possessed nearly the same atomic weights, like iron, cobalt, and nickel. Indeed, their names were suggested, on this supposition, with patriotic bias, as Anglium, Scotium, and Hibernium! But when the ratio of its specific heats had, at least in our opinion, unmistakably shown that it was molecularly monatomic, and not diatomic, as at first conjectured, it was necessary to believe that its atomic weight was 40, and not 20, and that it followed chlorine in the atomic table, and not fluorine. But here arises a difficulty. The atomic weight of chlorine is 35.5, and that of potassium, the next element in order in the table, is 39.1; and that of argon, 40, follows, and does not precede, that of potassium, as it might be expected to do. It still remains possible that argon, instead of consisting wholly of monatomic molecules, may contain a small percentage of diatomic molecules; but the evidence in favour of this supposition is, in my opinion, far from strong. Another possibility is that argon, as at first conjectured, may consist of a mixture of more than one element; but unless the atomic weight of one of the elements in the supposed mixture is very high, say 82, the case is not bettered, for one of the elements in the supposed trio would still have a higher atomic weight than potassium. And very careful experiments, carried out by Dr. Norman Collie and myself, on the fractional diffusion of argon, have disproved the existence of any such element with high atomic weight in argon, and, indeed, have practically demonstrated that argon is a simple substance, and not a mixture.

The discovery of helium has thrown a new light on this subject. Helium, it will be remembered, is evolved on heating certain minerals, notably those containing

uranium; although it appears to be contained in others in which uranium is not present, except in traces. Among these minerals are cleveite, monazite, fergusonite, and a host of similar complex mixtures, all containing rare elements, such as niobium, tantalum, yttrium, cerium, &c. The spectrum of helium is characterised by a remarkably brilliant yellow line, which had been observed as long ago as 1868 by Professors Frankland and Lockyer in the spectrum of the sun's chromosphere, and named "helium" at that early date.

The density of helium proved to be very close to 2.0, and, like argon, the ratio of its specific heat showed that it, too, was a monatomic gas. Its atomic weight therefore is identical with its molecular weight, viz., 4.0, and its place in the periodic table is between hydrogen and lithium, the atomic weight of which is 7.0.

The difference between the atomic weights of helium and argon is thus 36, or 40-4. Now there are several cases of such a difference. For instance, in the group the first member of which is fluorine we have—

Fluorine..	..	..	..	..	19	16.5
Chlorine..	..	..	..	..	35.5	19.5
Manganese ..	..	..	..	..	55	

In the oxygen group—

Oxygen ..	..	..	..	..	16	16
Sulphur ..	..	..	..	..	32	20.3
Chromium ..	..	..	..	..	52.3	

In the nitrogen group—

Nitrogen ..	..	..	..	..	14	17
Phosphorus ..	..	..	..	..	31	20.4
Vanadium ..	..	..	..	..	51.4	

And in the carbon group—

Carbon ..	..	..	..	..	12	16.3
Silicon ..	..	..	..	..	28.3	19.8
Titanium ..	..	..	..	..	48.1	

These instances suffice to show that approximately the differences are 16 and 20 between consecutive members of the corresponding groups of elements. The total differences between the extreme members of the short series mentioned are—

Manganese—Fluorine ..	..	..	..	36
Chromium—Oxygen ..	..	..	..	36.3
Vanadium—Nitrogen ..	..	..	..	37.4
Titanium—Carbon ..	..	..	..	36.1

This is approximately the difference between the atomic weights of helium and argon, 36.

There should, therefore, be an undiscovered element between helium and argon, with an atomic weight 16 units higher than that of helium, and 20 units lower than that of argon, namely 20. And if this unknown element, like helium and argon, should prove to consist of monatomic molecules, then its density should be half its atomic weight, 10. And pushing the analogy still farther, it is to be expected that this element should be as indifferent to union with other elements as the two allied elements.

My assistant, Mr. Morris Travers, has indefatigably aided me in a search for this unknown gas. There is a proverb about looking for a needle in a haystack; modern science, with the aid of suitable magnetic appliances, would, if the reward were sufficient, make short work of that proverbial needle. But here is a supposed unknown gas, endowed no doubt with negative properties, and the whole world to find it in. Still, the attempt had to be made.

We first directed our attention to the sources of helium—minerals. Almost every mineral which we could obtain was heated in a vacuum, and the gas which was evolved examined. The results are interesting. Most minerals give off gas when heated, and the gas contains, as a rule, a considerable amount of hydrogen, mixed with

carbonic acid, questionable traces of nitrogen, and carbonic oxide. Many of the minerals, in addition, gave helium, which proved to be widely distributed, though only in minute proportion. One mineral—malacone—gave appreciable quantities of argon; and it is noteworthy that argon was not found except in it (and, curiously, in much larger amount than helium), and in a specimen of meteoric iron. Other specimens of meteoric iron were examined, but were found to contain mainly hydrogen, with no trace of either argon or helium. It is probable that the sources of meteorites might be traced in this manner, and that each could be relegated to its particular swarm.

Among the minerals examined was one to which our attention had been directed by Professor Lockyer, named *eliasite*, from which he said that he had extracted a gas in which he had observed spectrum lines foreign to helium. He was kind enough to furnish us with a specimen of this mineral, which is exceedingly rare, but the sample which we tested contained nothing but undoubted helium.

During a trip to Iceland in 1895, I collected some gas from the boiling springs there; it consisted, for the most part, of air, but contained somewhat more argon than is usually dissolved when air is shaken with water. In the spring of 1896 Mr. Travers and I made a trip to the Pyrenees to collect gas from the mineral springs of Cauterets, to which our attention had been directed by Dr. Bouchard, who pointed out that these gases are rich in helium. We examined a number of samples from the various springs, and confirmed Dr. Bouchard's results, but there was no sign of any unknown lines in the spectrum of these gases. Our quest was in vain.

We must now turn to another aspect of the subject. Shortly after the discovery of helium, its spectrum was very carefully examined by Professors Runge and Paschen, the renowned spectroscopists. The spectrum was photographed, special attention being paid to the invisible portions, termed the "ultra-violet" and "infrared." The lines thus registered were found to have a harmonic relation to each other. They admitted of division into two sets, each complete in itself. Now, a similar process had been applied to the spectrum of lithium and to that of sodium, and the spectra of these elements gave only one series each. Hence, Professors Runge and Paschen concluded that the gas, to which the provisional name of helium had been given, was, in reality, a mixture of two gases, closely resembling each other in properties. As we know no other elements with atomic weights between those of hydrogen and lithium, there is no chemical evidence either for or against this supposition. Professor Runge supposed that he had obtained evidence of the separation of these imagined elements from each other by means of diffusion; but Mr. Travers and I pointed out that the same alteration of spectrum, which was apparently produced by diffusion, could also be caused by altering the pressure of the gas in the vacuum tube; and shortly after Professor Runge acknowledged his mistake.

These considerations, however, made it desirable to subject helium to systematic diffusion, in the same way as argon had been tried. The experiments were carried out in the summer of 1896 by Dr. Collie and myself. The result was encouraging. It was found possible to separate helium into two portions of different rates of diffusion, and consequently of different density by this means. The limits of separation, however, were not very great. On the one hand, we obtained gas of a density close to 2.0; and on the other, a sample of density 2.4 or thereabouts. The difficulty was increased by the curious behaviour, which we have often had occasion to confirm, that helium possesses a rate of diffusion too rapid for its density. Thus, the density of the lightest portion of the diffused gas, calculated from its rate of diffusion, was 1.874; but this corresponds to a real density of about 2.0. After our paper, giving an account of these experiments, had been

published, a German investigator, Herr A. Hagenbach, repeated our work and confirmed our results.

The two samples of gas of different density differ also in other properties. Different transparent substances differ in the rate at which they allow light to pass through them. Thus, light travels through water at a much slower rate than through air, and at a slower rate through air than through hydrogen. Now Lord Rayleigh found that helium offers less opposition to the passage of light than any other substance does, and the heavier of the two portions into which helium had been split offered more opposition than the lighter portion. And the retardation of the light, unlike what has usually been observed, was nearly proportional to the densities of the samples. The spectrum of these two samples did not differ in the minutest particular; therefore it did not appear quite out of the question to hazard the speculation that the process of diffusion was instrumental, not necessarily in separating two kinds of gas from each other, but actually in removing light molecules of the same kind from heavy molecules. This idea is not new. It had been advanced by Prof. Schützenberger (whose recent death all chemists have to deplore), and later, by Sir William Crookes, that what we term the atomic weight of an element is a mean; that when we say that the atomic weight of oxygen is 16, we merely state that the average atomic weight is 16; and it is not inconceivable that a certain number of molecules have a weight somewhat higher than 32, while a certain number have a lower weight.

We therefore thought it necessary to test this question by direct experiment with some known gas; and we chose nitrogen, as a good material with which to test the point. A much larger and more convenient apparatus for diffusing gases was built by Mr. Travers and myself, and a set of systematic diffusions of nitrogen was carried out. After thirty rounds, corresponding to 180 diffusions, the density of the nitrogen was unaltered, and that of the portion which should have diffused most slowly, had there been any difference in rate, was identical with that of the most quickly diffusing portion—i.e., with that of the portion which passed first through the porous plug. This attempt, therefore, was unsuccessful; but it was worth carrying out, for it is now certain that it is not possible to separate a gas of undoubted chemical unity into portions of different density by diffusion. And these experiments rendered it exceedingly improbable that the difference in density of the two fractions of helium was due to separation of light molecules of helium from heavy molecules.

(To be continued).

BACTERIOLOGICAL STUDY OF AMBERGRIS.

By H. BEAUREGARD.

I HAVE formerly shown, in concert with the regretted Professor G. Gouchet, that ambergris is an interesting calculus which is developed and has its seat in the rectum of the sperm whale.

This calculus, composed of crystals of ambrine mixed with a larger or smaller amount of black pigment derived from the rectal lining, contains also star-coral débris. When it is fresh, i.e., when it is just extracted from the rectum by the fishermen, it is of a soft consistency, and its odour is not at all agreeable on account of its predominant excrementitious character. But after being preserved for some years in an air-tight tin case it is gradually freed from this excrementitious odour, though losing little of its weight, and retains merely a delicate perfume *sui generis*, which gives it such a value that it reaches the price of from 3000 to 7000 frs. per kilogramme. This is not a case of slow desiccation, and cannot be imitated or accelerated by the withdrawal of water. The change is due to a microbe for which the author proposes

the name *Spirillum recti Physeteris*. As regards polymorphism this microbe is comparable to the spirillum of cholera. It is probable that the destruction of the fæcal odour and the genesis of the delicate perfume are microbial phenomena. It remains to determine if the spirillum in question is pathogenous, at least for terrestrial animals.—*Comptes Rendus*, cxxv., p. 254.

NOTICES OF BOOKS.

Twenty-first Annual Report of Her Majesty's Inspectors of Explosives; being their Annual Report for the Year 1896. Pp. 184. London: Eyre and Spottiswoode. 1897.

DURING the past year a further modification of the Explosives Act of 1875 has taken place, the Orders in Council relating to premises registered for mixed explosives having been repealed, and a new Order (No. 16) substituted. The main object of this new Order was to give relief in the storage of small-arm nitro-compounds.

The number of factories making explosives has had a further increase, five new ones having been started since 1895, while seven applications for new licenses are still under consideration. In the case of magazines there is a net increase of two, and there are nine further applications under consideration.

Every factory has been visited by the Inspectors at least once a year, and nearly one-half of them twice; and it is satisfactory to note that there has been no falling off in the high standard previously attained in the great majority of these establishments, while some which had been behind have been much improved.

A striking example of the great improvement in the conduct of the factories, since the passing of the Explosives Act, is to be found in the fact that only one death from accidents by fire or explosion in any factory has occurred during the year, and this in spite of the large increase in the number of factories, which is now 139, though three are temporarily closed.

In Appendix A will be found a table of all factories now in existence, with the classes of explosives authorised to be manufactured therein; and Appendix E gives their distribution about the country. There have been six additions to the list of authorised explosives during the year, particulars of which are set forth in Appendix D (1). As already explained in a previous Report, all nitro-cotton is now admitted to be explosive; collodion cotton, therefore, appears on the authorised list, but the exemptions—when it is in solution in alcohol and ether, or wet with methylated spirit, and enclosed in air-tight cases, still hold good.

Of the 358 samples taken during the year, and examined by Dr. Dupré, only 32 were rejected, and of these 9 were amorces.

One conviction has been obtained during the year for manufacturing fireworks containing sulphur and chlorate, contrary to the provisions of the Order in Council; one for manufacturing amorces and throw-downs with an excess of composition; while at two factories explosives have been placed under seizure. Although, as we mentioned above, there has been only one death from accident, there have been 46 accidents from one cause and another, by which in addition 26 persons were injured: details are to be found in Appendix W, and in the accident section of this Report.

It is satisfactory to see that no case of illegal manufacture has come under the notice of the Inspectors during the year.

There have been 416 visits paid during the year to the 384 magazines in the United Kingdom, and in every case the Inspectors were well satisfied, and there has been no accident by fire or explosion at any one of them during this period. There has been no fault found with the

character of the packing of explosives,—as a rule it has been excellent.

The amount of foreign nitro-glycerin compounds imported during 1896 shows a considerable increase over that imported in 1895; the increase being from 880,070 lbs. to 1,259,200 lbs., most of which was in carbonite: this explosive now heads the list, gelatin-dynamite no longer holding first place.

The Inspectors have no reason to modify their previously expressed opinion that their powers of search and seizure are ample and satisfactory. The total cases of seizure of all sorts was 15, the average for the previous ten years being 24.9.

As usual, the registered premises have been the most troublesome branch of the Inspector's duties; their number, their distribution, and the apparently extreme ignorance of the occupiers in many cases renders it difficult to make much impression on them as a mass,—still it is exceptional now to find large excesses of explosives stored in them.

A detailed list of all the accidents which came under the notice of the Inspectors is given; perhaps the most interesting of these was the one at the Arklow Factory on December 1st. A charge of glycerin was being nitrated in the ordinary way, and it is believed that about 60 lbs. of nitro-glycerin was in the nitrator. The acids had been blown in, and cooled, and the injection of glycerin had proceeded for about ten minutes, when suddenly, without warning, the charge fumed off. The connections to the drowning tank were immediately opened, but it was subsequently found that no nitro-glycerin was among the water and acids in the drowning tank, this leading to the conclusion that the whole of the 60 lbs. of nitro-glycerin had fumed off, in the few seconds from the first establishment of the action to the escape of the charge into the drowning tank; and as the thermometer did not show any increase of temperature—it stood at 12° C.—it is assumed that a leak in one of the coils had occurred and caused a local rise of temperature: such a leak was found after the accident, though it is confidently stated that the coils were tested by air pressure before the commencement of the operation, and that no leak existed then. Fortunately there was no loss of life or personal injury.

Among foreign explosions, one of the most tremendous of modern times occurred on the 19th of February, at Johannesburg, South Africa, when no less than 55 tons of blasting gelatin, contained in ten trucks, exploded on the railway, about 300 yards from Johannesburg station, under circumstances which in our opinion amount to criminal negligence, and almost inconceivable stupidity. The trucks containing the blasting gelatin had been standing for three and a half days, without supervision, in the blazing sun, in a place surrounded by human habitations; dynamite and detonators were carried in the same truck; the trucks were so badly packed that several cases of dynamite had fallen off in transit, and, further, this particular consignment was not packed in cases thick enough and strong enough according to the law. It is not so much a matter of surprise that, when a train that was being shunted ran into the trucks, owing to the points being wrongly turned, an explosion took place, as it is, that in this maze of ignorance and carelessness many other explosions had not already occurred.

A Course of Practical Chemistry. By M. M. PATTISON MUIR, M.A. Part I.—Elementary. Pp. 318. London: Longmans, Green, and Co. 1897.

THE author's experience as a teacher for twenty-five years has convinced him that qualitative analysis cannot be properly learned at an early stage of a course of practical chemistry. He therefore, in this volume, lays more stress on the simple work of preparations, and experiments on the reactions of the chief inorganic compounds, before going on to actual analysis. This is as it should

be; an apprentice to a carpenter learns to saw straight before he begins to make a cabinet.

The volume now before us (Part I.) is divided into three sections, and these again are subdivided into fifty-two lessons.

Section I. deals with experiments on chemical change; preparations of various compounds; and the reactions of acids, alkalies, and salts—in which all the ordinary elementary work of a chemical laboratory is fully described. There are also some notes preceding the actual lessons giving useful hints to a beginner, straight and to the point. *E.g.*, "Note II. Always read the whole of the directions before beginning an experiment." "When you are told to 'heat gently,' this means use a small Bunsen flame for heating, and keep the flame at some distance from the apparatus to be heated."

Section II. concerns volumetric estimations of acids, alkalies, iron salts, chlorides, iodine, &c., the necessary standard solutions being given.

In Section III. we arrive at qualitative analysis, beginning with experiments to illustrate the methods used for dividing the metals into groups, and distinguishing the several metals in these same groups, going all through the six groups, and finally coming to the examination of a solid by dry or blowpipe tests.

The Appendices, of which there are five, contain, amongst other information, instructions for making simple apparatus, the usual tables, list of reagents and their preparation, standard solutions, and a list of substances suitable for the various exercises. The Index is good and complete, and the whole arrangement of the book seems to be very convenient.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 3, July 19, 1897.

The Minister of Public Instruction communicated the text of the decree by which the President of the French Republic approves the election of Prof. Virchow as a Foreign Associate *vice* M. Tchebicheff, deceased.

Researches on the State of Elements other than Carbon in Cast-irons and Steels.—Ad. Carnot and M. Goutal.—Will be inserted in full.

Phenomenon of the Electric Arc.—A. Blondel.—This paper requires the accompanying diagram.

Action of Electric Charges on the property of Discharge created by the X Rays in the Air.—E. Villari.—It is known that gases traversed by the X rays acquire the property of discharging electrified conductors. I have demonstrated that they retain this property, though in a less degree after having traversed tubes of glass or of lead of 20 metres in length, or even upwards. I have arrived at the following conclusions:—Air traversed by the X rays and blown against the end of a wire in its natural state retains entirely its discharging power undiminished. If blown against the end of an electrified wire (+) it entirely loses its power of discharging an electroscope (±) having the same sign as that of the wire. Air traversed by the X rays and drawn against the approximating ends of two wires having opposite charges loses all discharging power. The air traversed by X rays, on passing by an ozoniser, acts in these experiments as if its mols. had opposite charges by which it can discharge electrified bodies.

Properties of Gases Traversed by the X Rays, and on the Properties of Luminescence on Photographic Bodies.—G. Sagnac.

The Spectrum of Carbon.—A. de Gramont.—This paper requires the accompanying diagram.

Action of Cupric Hydrate upon Solutions of Silver Nitrate and Basic Argentic Cuprate.—Paul Sabatier.

—The formation of a basic mixed argento-cupric salt is not confined to the nitrates. An analogous production ensues on setting out from the sulphates, chlorates, and hyposulphates, as the author intends to show in a future communication.

Hydrobenzamide, Amarine, and Lophine.—Marcel Delépine.—An elaborate thermo-chemical memoir, in which we recognise the constant universality of the thermic laws regulating the chemical transformations of the complicated molecules.

New Syntheses by means of Cyano-succinic Ether.—L. Barth.—Not adapted for useful abstraction.

Certain Compounds of Phenylhydrazine and Metallic Nitrates.—J. Moitessier.—The nitrates of the magnesian metals combine directly with phenylhydrazine like the corresponding haloid salts, yielding crystalline compounds presenting the reactions of phenylhydrazine and those of the metals which they contain. They deflagrate if heated, as do the nitrates in presence of carbon, leaving a more or less abundant residue of metallic oxide.

On the Aloines.—E. Léger.—The aloines may be divided into two groups: the first including barbaloine, socaloine, zanaloin, and curaraloin; whilst the second contains a single representative, mataloin.

MISCELLANEOUS.

Action of X Rays upon the Temperature of Animals.—L. Lecerclé.—Exposure to X rays modifies the cutaneous and rectal temperatures both in the same direction. Under their influence these temperatures sink at first, and then rise above the initial degree.—*Comptes Rendus*, cxxv., No. 4.

Actino-Electric Effects of the Röntgen Rays.—S. Puggenheimer.—The author has obtained the following results:—If we plunge two identical electrodes into a liquid and expose the *n*-th one to the X rays there is set up a current which ordinarily flows from the plate exposed to the X rays to the *n*-th of the external circuit. The intensity of the current depends on the intensity of the radiation.—*Comptes Rendus*, cxxv., No. 1.

On the Applications of Electrolysis to Organic Chemistry.—L. Gourwitsch.—The action of electrolysis on the salts of the fatty acids nearly always gives rise to the formation of alcohols, ethers, acids, &c., in quantities variable according to the conditions of the experiment. A large number of experiments made by Loeb, Bourgoin, Kolbe, Brown and Walker, Mulliken, and many others, are quoted, and the "mechanism" of the reactions are discussed. Iodoform is now being prepared electrolytically by substitution, by passing the current through a solution of iodide of potassium in alcohol or aqueous acetone, and neutralising the excess of potash formed by carbonic acid; the iodine and the potash formed by the action of the current react with the solvent, and form crystals of perfectly pure iodoform. The nitrified derivatives of the aromatic series serve best for studying the reduction by electrolysis. In 1882 Kendall patented the manufacture of aniline and toluidine by the action of the electric current on mixtures of nitrobenzene and nitrotoluene with concentrated sulphuric acid; but the return was very bad, and the process was of no practical value. Twelve years later the subject was again taken up, and it was shown that aniline was formed even in acid solution. It is interesting to note that the platinum electrodes were strongly attacked in the experiments on carbamate of ammonia, a complicated platino-ammoniacal base being formed.—*Moniteur Scientifique*, xi., Part 1.

Australasian Association for the Advancement of Science.—The objects of this Association, which is now about to commence its Seventh Session, are to give a stronger impulse and a more systematic direction to scientific enquiry, and to promote the intercourse of those who cultivate science in different parts of the Australasian Colonies and in other countries. The Seventh Session will commence in Sydney, on Monday, January 6th next, and will last till the next Thursday, under the presidency of Professor A. Liversidge, M.A., LL.D., F.R.S. The work of the Association will be divided into ten sections, devoted to all the principal scientific subjects. The subscription to become an Interim Member is £1, or £10 to become a Life Member. Excursions will be organised to places of interest, such as the various mining districts, the Zenolan and Wambyan Caves, the Blue Mountains, &c. All particulars can be obtained from the Hon. Secretaries, Australasian Association for the Advancement of Science, The University, Sydney.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Cooling Water.—Can any reader give me particulars of a simple, cheap, and effective means of cooling (and the colder the better) a fairly large quantity of water, say three or four pails full, for use in a hot climate? The water is not for cooking or washing purposes.—D. J. P.

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ADDRESS TO THE CHEMICAL SECTION OF THE BRITISH ASSOCIATION. TORONTO, 1897.

By Professor WILLIAM RAMSAY, Ph.D., LL.D., Sc.D., F.R.S.,
President of the Section.

(Concluded from p. 93).

THE apparatus used for diffusion had a capacity of about two litres. It was filled with helium, and the operation of diffusion was carried through thirty times. There were six reservoirs, each full of gas, and each was separated into two by diffusion. To the heavier portion of one lot, the lighter portion of the next was added, and in this manner all six reservoirs were successively passed through the diffusion apparatus. This process was carried out thirty times, each of the six reservoirs having had its gas diffused each time, thus involving 180 diffusions. After this process, the density of the more quickly diffusing gas was reduced to 2.02, while that of the less quickly diffusing had increased to 2.27. The light portion on re-diffusion hardly altered in density, while the heavier portion, when divided into three portions by diffusion, showed a considerable difference in density between the first third and the last third. A similar set of operations was carried out with a fresh quantity of helium, in order to accumulate enough gas to obtain a sufficient quantity for a second series of diffusions. The more quickly diffusing portions of both gases were mixed and re-diffused. The density of the lightest portion of these gases was 1.98; and after other 15 diffusions, the density of the lightest portion had not decreased. The end had been reached; it was not possible to obtain a lighter portion by diffusion. The density of the main body of this gas is therefore 1.98; and its refractivity, air being taken as unity, is 0.1245. The spectrum of this portion does not differ in any respect from the usual spectrum of helium.

As re-diffusion does not alter the density or the refractivity of this gas, it is right to suppose that either one definite element has now been isolated, or that, if there are more elements than one present, they possess the same, or very nearly the same, density and refractivity. There may be a group of elements, say three, like iron, cobalt, and nickel; but there is no proof that this idea is correct, and the simplicity of the spectrum would be an argument against such a supposition. This substance, forming by far the larger part of the whole amount of the gas, must, in the present state of our knowledge, be regarded as pure helium.

On the other hand, the heavier residue is easily altered in density by re-diffusion, and this would imply that it consists of a small quantity of a heavy gas mixed with a large quantity of the light gas. Repeated re-diffusion convinced us that there was only a very small amount of the heavy gas present in the mixture. The portion which contained the largest amount of heavy gas was found to have the density 2.275, and its refractive index was found to be 0.1333. On re-diffusing this portion of gas until only a trace sufficient to fill a Plücker's tube was left, and then examining the spectrum, no unknown lines could be detected, but, on interposing a jar and spark gap, the well-known blue lines of argon became visible; and even without the jar the red lines of argon and the two green groups were distinctly visible. The amount of

argon present, calculated from the density, was 1.64 per cent, and from the refractivity 1.14 per cent. The conclusion had therefore to be drawn that the heavy constituent of helium, as it comes off the minerals containing it, is nothing new, but, so far as can be made out, merely a small amount of argon.

If, then, there is a new gas in what is generally termed helium, it is mixed with argon, and it must be present in extremely minute traces. As neither helium nor argon has been induced to form compounds, there does not appear to be any method, other than diffusion, for isolating such a gas, if it exists, and that method has failed in our hands to give any evidence of the existence of such a gas. It by no means follows that the gas does not exist; the only conclusion to be drawn is that we have not yet stumbled on the material which contains it. In fact, the haystack is too large and the needle too inconspicuous. Reference to the periodic table will show that between the elements aluminium and indium there occurs gallium, a substance occurring only in the minutest amount on the earth's surface; and following silicon, and preceding tin, appears the element germanium, a body which has as yet been recognised only in one of the rarest of minerals, argyrodite. Now, the amount of helium in fergusonite, one of the minerals which yields it in reasonable quantity, is only 33 parts by weight in 100,000 of the mineral; and it is not improbable that some other mineral may contain the new gas in even more minute proportion. If, however, it is accompanied in its still undiscovered source by argon and helium, it will be a work of extreme difficulty to effect a separation from these gases.

In these remarks it has been assumed that the new gas will resemble argon and helium in being indifferent to the action of reagents, and in not forming compounds. This supposition is worth examining. In considering it, the analogy with other elements is all that we have to guide us.

We have already paid some attention to several triads of elements. We have seen that the differences in atomic weights between the elements fluorine and manganese, oxygen and chromium, nitrogen and vanadium, carbon and titanium, is in each case approximately the same as that between helium and argon, viz., 36. If elements further back in the periodic table be examined, it is to be noticed that the differences grow less, the smaller the atomic weights. Thus, between boron and scandium the difference is 33; between beryllium (glucium) and calcium, 31; and between lithium and potassium, 32. At the same time, we may remark that the elements grow liker each other, the lower the atomic weights. Now, helium and argon are very like each other in physical properties. It may be fairly concluded, I think, that in so far they justify their position. Moreover, the pair of elements which show the smallest difference between their atomic weights is beryllium and calcium; there is a somewhat greater difference between lithium and potassium. And it is in accordance with this fragment of regularity that helium and argon show a greater difference. Then again, sodium, the middle element of the lithium triad, is very similar in properties both to lithium and potassium; and we might, therefore, expect that the unknown element of the helium series should closely resemble both helium and argon.

Leaving now the consideration of the new element, let us turn our attention to the more general question of the atomic weight of argon, and its anomalous position in the periodic scheme of the elements. The apparent difficulty is this:—The atomic weight of argon is 40; it has no power to form compounds, and thus possesses no valency; it must follow chlorine in the periodic table, and precede potassium; but its atomic weight is greater than that of potassium, whereas it is generally contended that the elements should follow each other in the order of their atomic weights. If this contention is correct, argon should have an atomic weight smaller than 40.

Let us examine this contention. Taking the first row of elements, we have:—

Li=7, Be=9.8, B=11, C=12, N=14, O=16, F=19,
? = 20.

The differences are:—

2.8, 1.2, 1.0, 2.0, 2.0, 3.0, 1.0.

It is obvious that they are irregular. The next row shows similar irregularities. Thus:—

(? = 20, Na = 23, Mg = 24.3, Al = 27, Si = 28, P = 31, S = 32,
Cl = 35.5, A = 40.

And the differences:—

3.0, 1.3, 2.7, 1.0, 3.0, 1.0, 3.5, 4.5.

The same irregularity might be illustrated by a consideration of each succeeding row. Between argon and the next in order, potassium, there is a difference of -0.9; that is to say, argon has a higher atomic weight than potassium by 0.9 unit, whereas it might be expected to have a lower one, seeing that potassium follows argon in the table. Farther on in the table there is a similar discrepancy. The row is as follows:—

Ag = 108, Cd = 112, In = 114, Sn = 119, Sb = 120.5,
Te = 127.7, I = 127.

The differences are:—

4.0, 2.0, 5.0, 1.5, 7.2, -0.7.

Here, again, there is a negative difference between tellurium and iodine. And this apparent discrepancy has led to many and careful re-determinations of the atomic weight of tellurium. Professor Brauner, indeed, has submitted tellurium to methodical fractionation, with no positive results. All the recent determinations of its atomic weight give practically the same number, 127.7.

Again, there have been almost innumerable attempts to reduce the differences between the atomic weights to regularity, by contriving some formula which will express the numbers which represent the atomic weights, with all their irregularities. Needless to say, such attempts have in no case been successful. Apparent success is always attained at the expense of accuracy, and the numbers reproduced are not those accepted as the true atomic weights. Such attempts, in my opinion, are futile. Still, the human mind does not rest contented in merely chronicling such an irregularity; it strives to understand why such an irregularity should exist. And, in connection with this, there are two matters which call for our consideration. These are:—Does some circumstance modify these "combining proportions" which we term "atomic weights"? And is there any reason to suppose that we can modify them at our will? Are they true "constants of Nature," unchangeable, and once for all determined? Or are they constant merely so long as other circumstances, a change in which would modify them, remain unchanged?

In order to understand the real scope of such questions, it is necessary to consider the relation of the "atomic weights" to other magnitudes, and especially to the important quantity termed "energy."

It is known that energy manifests itself under different forms, and that one form of energy is quantitatively convertible into another form, without loss. It is also known that each form of energy is expressible as the product of two factors, one of which has been termed the "intensity factor," and the other the "capacity factor." Professor Ostwald, in the last edition of his "*Allgemeine Chemie*," classifies some of these forms of energy as follows:—(see next column).

In each statement of factors the "capacity factor" is placed first, and the "intensity factor" second.

In considering the "capacity factors" it is noticeable that they may be divided into two classes. The two first kinds of energy, kinetic and linear, are *independent of the nature of the material* which is subject to the energy. A mass of lead offers as much resistance to a given force, or, in other words, possesses as great inertia, as an equal mass of hydrogen. A mass of iridium, the densest solid,

Kinetic energy is the product of Mass into the square of velocity.

Linear " " Length into force.

Surface " " Surface into surface tension.

Volume " " Volume into pressure.

Heat " " Heat-capacity (entropy) into temperature.

Electrical " " Electric capacity into potential.

Chemical " " "Atomic weight" into affinity.

counterbalances an equal mass of lithium, the lightest known solid. On the other hand, surface energy deals with molecules, and not with masses. So does volume energy. The volume energy of 2 grms. of hydrogen, contained in a vessel of 1 litre capacity, is equal to that of 32 grms. of oxygen at the same temperature, and contained in a vessel of equal size. Equal masses of tin and lead have not equal capacity for heat; but 119 grms. of tin has the same capacity as 207 grms. of lead,—that is, equal atomic masses have the same heat capacity. The quantity of electricity conveyed through an electrolyte under equal difference of potential is proportional, not to the mass of the dissolved body, but to its equivalent,—that is, to some simple fraction of its atomic weight. And the capacity factor of chemical energy is the atomic weight of the substance subjected to the energy. We see, therefore, that while mass or inertia are important adjuncts of kinetic and linear energies, all other kinds of energy are connected with atomic weights, either directly or indirectly.

Such considerations draw attention to the fact that quantity of matter (assuming that there exists such a carrier of properties as we term "matter") need not necessarily be measured by its inertia, or by gravitational attraction. In fact, the word "mass" has two totally distinct significations. Because we adopt the convention to measure quantity of matter by its mass, the word "mass" has come to denote "quantity of matter." But it is open to anyone to measure a quantity of matter by any other of its energy factors. I may, if I choose, state that those quantities of matter which possess equal capacities for heat are equal; or that "equal numbers of atoms" represent equal quantities of matter. Indeed, we regard the value of material as due rather to what it can do, than to its mass; and we buy food, in the main, on an atomic, or perhaps a molecular, basis, according to its content of albumen. And most articles depend for their value on the amount of food required by the producer or the manufacturer.

The various forms of energy may therefore be classified as those which can be referred to an "atomic" factor, and those which possess a "mass" factor. The former are in the majority. And the Periodic Law is the bridge between them; as yet, an imperfect connection. For the atomic factors, arranged in the order of their masses, display only a partial regularity. It is undoubtedly one of the main problems of physics and chemistry to solve this mystery. What the solution will be is beyond my power of prophecy; whether it is to be found in the influence of some circumstance on the atomic weights, hitherto regarded as among the most certain "constants of Nature"; or whether it will turn out that mass and gravitational attraction are influenced by temperature, or by electrical charge, I cannot tell. But that some means will ultimately be found of reconciling these apparent discrepancies, I firmly believe. Such a reconciliation is necessary, whatever view be taken of the nature of the universe and of its mode of action; whatever units we may choose to regard as fundamental among those which lie at our disposal.

In this address I have endeavoured to fulfil my promise to combine a little history, a little actuality, and a little

prophecy. The history belongs to the Old World; I have endeavoured to share passing events with the New; and I will ask you to join with me in the hope that much of the prophecy may meet with its fulfilment on this side of the Ocean.

A POSSIBLE NEW ELEMENT, OR POSSIBLE NEW ELEMENTS, IN CAST-IRON AND BLAST-FURNACE BOILER-DUST.

By G. G. BOUCHER.

It will appear at first somewhat surprising to those accustomed to make analyses of cast-iron that there exists in that metal another element besides the elements C, Si, S, P, Mn, Cu, As, Sb, Cr, W, Ti, Ni, Co, Al, K, Na, Mg, Ca, Li, and V, already known to be present in cast-iron. There does, however, appear to be another metal present, and in this article I have attempted to describe a metal which I discovered in the iron and boiler-dust made at these works. I have described it as a possible new element, because as yet it has not been examined spectroscopically, and, although its properties are unlike those of any metal I know, it is possible that it may be one of those compounds so difficult to separate by chemical means, the properties of which can only be determined by means of the spectroscope. I thought it better, therefore, not to call it a new element till the result of the spectrum analysis is known. A small quantity of the metal is now in the hands of Sir William Crookes, to whom I wrote describing my discovery, and requesting that he would take a little for analysis. He very kindly consented to do so, and I hope in a short time to be able to give the result of his analysis. In the meantime I have written this article hoping that it may prove interesting.

To prepare the metal it is necessary to take large quantities of iron, as the metal is present in very small quantities. On four different occasions I obtained 0.0033 per cent, 0.0027 per cent, 0.0053 per cent, and 0.0060 per cent of the metal, using 100 grms. of iron in each case. An analysis of an average sample of iron ore only yielded 0.0019 per cent of the metal.

To obtain the metal, 100 grms. of iron are weighed out into a large beaker capable of holding about 3 litres; 2000 c.c. of dilute H_2SO_4 (1 acid to 5 H_2O) are run in, the beaker is covered with a clock glass, placed on a warm plate, and the iron completely dissolved. The beaker is then removed, the contents allowed to cool, H_2S is passed through the solution to precipitate any small quantities of metal which may have dissolved, and it is then allowed to stand till all the graphite, &c., have settled. The clear liquid is poured off; the graphite, &c., are filtered into a quick filter-paper, and thoroughly washed with hot water till free from iron. The graphite, SiO_2 , As, Sb, Cu, and unknown metal collected on the paper, are washed off into a beaker and treated with 4 or 5 grms. of KClO_3 and 50 c.c. HCl . The beaker is placed on a hot plate and gradually heated to boiling, and boiled till all free Cl is evolved. The metals being converted into chlorides, the silica and graphite are filtered off, well washed with hot water, and the filtrate saturated with H_2S . The precipitate—which consists of the sulphides of As, Sb, Cu, and unknown metal—is allowed to settle, then filtered, and well washed with H_2S water. The sulphides with filter-paper are placed in a small beaker, HCl is added, and about 2 grms. KClO_3 ; the beaker is placed on a warm plate, and when the sulphides are completely dissolved and the free chlorine driven off, the solution is filtered, the bulk of the liquid being kept as small as possible. The As is precipitated by MgCl_2 , AmHO , and AmCl , the solution being allowed to stand with repeated stirring about twelve hours. When the As is entirely precipitated it is filtered off, and the solution saturated with H_2S to

precipitate the Cu; the CuS is allowed to settle, filtered off, and the solution made slightly acid with HCl . The Sb and unknown metal are precipitated as sulphides, the precipitate being as a rule of a brown colour. The sulphides are allowed to settle, and when the liquid is quite clear it is poured off; 30 c.c. HCl (1 acid to 2 H_2O) are poured on to the sulphide, and the solution boiled. The precipitate is allowed to settle, the clear liquid poured off, and another 30 c.c. dilute HCl are added; the solution is boiled again, and the precipitate then allowed to settle. This is repeated three or four times, till all the Sb_2S_3 is completely dissolved. The unknown metal remains behind as a heavy dark brown sulphide, it being almost insoluble in boiling dilute HCl (1 to 1). This sulphide is thoroughly washed with hot water, and then dissolved in a dilute solution of KHO ; the solution is saturated with H_2S , and filtered from any small quantities of CuS or other sulphides formed. The filtrate is made slightly acid with HCl and the brown sulphide precipitated filtered off; it is again dissolved in KHO , the solution saturated with H_2S , and filtered. HCl is added to precipitate the sulphide; it is filtered on to a paper, and thoroughly washed with hot water. This process is repeated till the KHO solution of the sulphide produces no precipitate with H_2S . The brown metallic sulphide—which should now be absolutely free from As, Sb, Sn, Bi, and Cu—is thrown on to a filter-paper, thoroughly washed with hot distilled water, and dissolved in hot HNO_3 (1 to 2). The solution is filtered, and made just alkaline with AmHO to precipitate, or make doubly sure of the absence of, Sb, Sn, Bi, and Te. These metals are, however, hardly likely to be present. The metal is again precipitated as sulphide, filtered off, well washed with hot water, and dried in the water-oven. When quite dry the precipitate is brushed off the filter-paper into a porcelain crucible, and placed in a muffle which is hardly red-hot. The sulphide burns to an oxide of a pale yellow colour, which melts very easily, and at a full red-heat completely vapourises; hence it is necessary to keep the crucible at a very low red-heat, and to watch this part of the process very carefully. The oxide can also be obtained by dissolving the sulphide in HNO_3 , evaporating to dryness, and heating gently as described before, but it is not in such a good condition for reduction as that obtained by oxidation in a muffle. The metal can be obtained from the oxide either by heating it in a current of hydrogen or by fusing it with pure KCN .

The metal so obtained is a black powder, slightly soluble in cold strong HCl and H_2SO_4 , and very little more soluble on boiling in these acids. It is soluble in dilute and strong HNO_3 , and very easily soluble in aqua regia. It is insoluble in dilute HCl and H_2SO_4 .

Heated in a current of air the metal glows, and is converted into the yellow oxide, part of which vapourises and condenses on the side of the tube.

The oxide so formed, as I have said before, melts at a low temperature, and when allowed to cool crystallises out in long transparent pointed crystals, which are very beautiful when examined through the microscope. If a small quantity of the oxide be placed in a covered porcelain crucible and heated to a full red heat for one or two minutes in a muffle, on taking it out and examining it the crucible side will be found to be covered with long transparent colourless crystals. I have in my possession crystals prepared in this manner over a quarter of an inch long. The first lot of crystals I obtained were very nearly half an inch long and almost completely filled the crucible; they were quite transparent and appeared to be capable of refracting light.

The oxide, heated with borax, gives in the outer flame a clear colourless bead, and in the reducing flame a light-coloured pink bead. Heated with microcosmic salt it gave a clear chrome-green bead, both in oxidising and reducing flame; the bead obtained in the reducing flame being considerably darker in colour than that obtained in the oxidising flame. Fused with Na_2CO_3 it gave a colourless mass completely soluble in water. The oxide is difficultly

soluble in HCl, almost insoluble in H_2SO_4 , and insoluble in HNO_3 .

WET REACTIONS.

Chloride of the Metal.

Reagent.	Observation.
HCl	No precipitate.
H_2S	A dark brown precipitate from slightly acid solutions, soluble in SAM_2 , S_2Am_2 , Na_2S , KHO , and NaHO ; insoluble in boiling dilute HCl (1 to 1) and H_2SO_4 ; soluble in aqua regia and HNO_3 .
NaHO , KHO , AmHO	No precipitate, but slight blue colouration of the solution.
$\text{Na}_2\text{S}_2\text{O}_3$	A violet colouration, which turns brown on heating with a few drops of HCl ; the metal being precipitated as sulphide.
Na_2SO_3	No precipitate or apparent change even on boiling.
SnCl_2	No precipitate or apparent change even on boiling.
FeSO_4	No precipitate or apparent change even on boiling.
BaCO_3 , $(\text{Am})_2\text{CO}_3$	No precipitate.
CaCO_3 , Na_2CO_3 ..	" "
Magnesia mixture (AmCl and AmHO)	" "
AmCl , KCl	" "
Zn , Fe	A fine black deposit of metal. Part of the metal is evolved in combination with hydrogen. On lighting the issuing gas at a jet and placing a cold porcelain lid in the flame, the metal is deposited. The deposit is almost black and has very little lustre. It is insoluble in HCl , and a freshly prepared solution of bleaching powder has no action on it.
K_2CrO_4	No precipitate.
KCN	" "
Na_2HPO_4 and $\text{NaC}_2\text{H}_3\text{O}_2$	" "
K_3FeCy_6	" "
K_4FeCy_6	A dark brown flocculent precipitate from neutral solutions. Soluble in acids and alkalis.
H_2SO_4 , HCl	The most characteristic reaction for this metal appears to be that produced by the addition of a few drops of H_2SO_4 to the chloride or nitrate. On evaporating down till fumes of H_2SO_4 are driven off, and allowing to cool, the liquid assumes after a short time a most magnificent blue colour. Very minute quantities of the metal can be detected by this means. The same colour is produced by evaporating down two or three times with HCl to very nearly dryness. The colour is entirely destroyed by the addition of water.

The Metal in Boiler Dust.

A similar metal in every respect but one I have also found in the boiler dust. It has the same appearance, forms an oxide to all appearances the same as the other, and produces the same chemical changes with the reagents given with the exception of SnCl_2 . This reagent produces a dark blue colour, which on boiling with a little HCl turns to a dark brown.

It is almost more difficult to obtain this metal from

boiler dust than it is to obtain the metal from iron, as it is present in such minute quantities. To obtain about 0.3 grm. of the metal I had to treat quite a ton of dust, and as this was done in small quantities, taking about 28 lbs. at a time, it required considerable time, patience, and labour. It is not always possible to obtain the metal, as at times it seems to disappear altogether; at one time I was unable to find it for quite two months. It also seems to be present only in the dust from a few of the boilers. The lighter dust does not appear to contain any of the metal, it being only in heavy and dark coloured dust that I have been able to find it.

To prepare the metal, about 28 lbs. of dust are placed in a large canvas filter and well washed with water, the washings being allowed to run into a Winchester quart bottle. To save using a large quantity of water, the washings are run through several times till they are saturated with the salts K_2SO_4 and Na_2SO_4 , which are present in large quantities. If the metal is present the solution generally has a dark yellow colour. H_2S is passed into this solution for about ten or fifteen minutes, and then about 50 c.c. strong HCl are run in and the bottle thoroughly shaken. The metal is precipitated as sulphide, and in about twelve hours settles at the bottom of the bottle. The clear liquid is syphoned off, and the sulphide washed into a large stoppered bottle, there to wait till many more such precipitates have been added to it.

When a sufficient quantity has been collected, it is filtered off, washed into a large porcelain basin, and dissolved in aqua regia. 15 c.c. H_2SO_4 are added, and the solution is evaporated down till fumes of H_2SO_4 appear. The solution is allowed to cool, water is added, and it is then filtered from the insoluble residue of PbSO_4 , SiO_2 , S , &c. The filtrate is made alkaline with AmHO and saturated with H_2S . The CuS , Bi_2S_3 , &c., are filtered off, washed with SAM_2 water, and the solution is acidified with HCl to precipitate the metal. It is filtered on to a paper, well washed with hot water, afterwards dissolved in KHO , and treated as described before, under the preparation of the metal from iron.

A glance at the reactions of these metals will show that there is some reason to believe that there is something new about them—as will be easily seen from a comparison of the reactions of these metals and those which they seem to most closely resemble. It is more than probable that these metals will turn out to be the same, but whether spectrum analysis will show them to be metals already discovered remains to be seen. I hardly think they can be. These metals are, however, very interesting, inasmuch as I believe they have not been discovered in cast-iron or boiler dust before.

It may be interesting to know that I also found in the boiler dust the metals thallium, cadmium, zinc, lead, bismuth, arsenic, and antimony.

North Lonsdale Iron and Steel Co.,
Ulverston, August 18, 1897.

NITROGEN IN ANALYSES.*

By C. F. JURITZ, M.A.,
Senior Analyst, Government Laboratory, Cape Town.

THE ordinary farmer is no doubt much perplexed at the wide range of figures expressing the percentages of nitrogenous constituents, when comparing the results of analyses by different authors, of articles regarding whose composition he is desirous of learning something.

Comparing one fertiliser with another of the same class, such as bone meal, for example, one authority may tell him that it contains 3½ per cent of *nitrogen*, another that it contains 20 per cent of *nitrogenous matter*, while

* Abridged from the *Agricultural Journal*, Cape of Good Hope, June 10, 1897.

a third might possibly return it as containing $4\frac{1}{2}$ per cent of ammonia.

Suppose a farmer is using linseed cake, and wishes to compare results. He finds one cake contains 27 per cent of albuminous compounds, while another has only $4\frac{3}{4}$ per cent of nitrogen. "What a difference!" he says, thinking that the same substance is referred to in each case. Again, he finds that wheat contains 12 per cent of nitrogenous compounds, while peas do not contain more than 4 per cent of nitrogen; this puzzles him, because he has always understood that peas were of the "nitrogen-loving" sort.

It is really a very simple matter to reconcile these seeming differences. As 17 parts of ammonia contain 14 parts of nitrogen, we know that 17 per cent of ammonia is equivalent to 14 per cent of nitrogen. Again, the terms "nitrogenous matter," "albumenoids," &c., are frequently used interchangeably for a certain class of substances which contain about 16 per cent of nitrogen; that is, 1 part of nitrogen in $6\frac{1}{2}$ parts of nitrogenous matter. If, therefore, we have a bone-meal containing $3\frac{1}{2}$ per cent of nitrogen; to find how much ammonia this is equal to, we must multiply by 17 and divide by 14. The amount of nitrogenous matter is found by multiplying the nitrogen by $6\frac{1}{2}$ and *vice versa*.

Now, taking the first instance quoted, viz., the three samples of bone-meal—The first contained $3\frac{1}{2}$ per cent of nitrogen; the second 20 per cent of nitrogenous matter—that is, $\frac{20 \times 16}{100} = 3\cdot2$ per cent of nitrogen—or really

less than No. 1. The third shows $4\frac{1}{2}$ per cent of ammonia, or $\frac{4\frac{1}{2} \times 14}{17} = 3\cdot5$ per cent of nitrogen. Similarly

with the two samples of linseed cake; the latter contains more nitrogen than the former, being $4\frac{3}{4} \times 6\frac{1}{2}$, or practically 30 per cent.

As a matter of fact, most text-books explain their results by a footnote, as in the following analysis of hay:—

Moisture	16·50	per cent
*Nitrogenous substances ..	15·81	"
Carbonaceous principles ..	37·63	"
Woody fibre.. .. .	22·47	"
Mineral matter	6·59	"
	99·00	

* Containing nitrogen, 2·53 (equal to ammonia, 2·08 per cent).

EXPERIMENTAL RESEARCHES ON GLASSES,

CARRIED OUT UNDER THE DIRECTION OF THE
"COMMITTEE OF CHEMICAL ARTS," OF THE SOCIÉTÉ
DE L'ENCOURAGEMENT.*

By L. GRENET.

THE object of these researches, for which the proprietor of the glass-works at Blanzay, M. Solvay, and the Company of Saint-Gobain, kindly offered the use of their works to the Committee, was to determine the relations existing between the chemical composition of glass and its most important properties, such as dilatation, fusibility, tenacity, refrangibility, and alterability. The first part, which concerns dilatation, and has been done in conjunction with M. Chatenet, is now finished, and forms the subject of this article.

Very little work has till now been done on the dilatation of glass—that of Fizeau, Schott, and Damour being all that is worth recalling.

The tension of an enamel applied to a body less dilatable is easily found by the following equation:—

$$1 - \Delta (T - t) = [1 - \delta (T - t)] \left(\frac{R}{E} + 1 \right)$$

when Δ = the coefficient of dilatation of the body to be enamelled;

δ . Coefficient of dilatation of the enamel;

T. The temperature beyond which the enamel must not be raised without becoming permanently deformed;

t. The lowest temperature to which the enamelled body is reduced;

R. Tension of the enamel;

E. Coefficient of elasticity of the enamel.

Compared with metals the dilatation of enamel is almost always too feeble, and if the tension due to the difference of dilatation is too great the adherence of the enamel to the metal may be insufficient to resist it, and the enamel will crack off; but it is better, if the coefficients of dilatation cannot be made to agree, that that of the enamel should be the lesser, so that it remains under compression.

The measurements in all these experiments were made by M. Fizeau's method. The apparatus used was very completely described by M. Damour, modified by M. le Chatelier, in the *Bulletin* of February, 1896: the only important modification since this time has been the substitution of warming by means of water instead of by air. It consists of three points of tempered steel, which serve as a support for the glass under examination. Parallel to the base of this tripod, and 2 c.m. from it, are three levelling screws of hardened steel. The glass, cut into a prism of 2 or 3 c.m. high and polished on its lower surface, is placed on the tripod, and adjusted by means of the screws in such a manner that its polished face coincides almost exactly with the base of the tripod.

Under these conditions, if we place this over a bi-convex lens, and illuminate with monochromatic light, we can observe Newton's rings. The prism and its support on the bi-convex lens are placed on the top of a vertical tube, at the lower end of which two prisms are arranged, at its junction with a horizontal tube. The yellow light from a Bunsen is received on and totally reflected up by a prism, to the glass under experiment; from there it is reflected down the vertical tube again on to another prism fitted with a lens which again reflects it further along the horizontal tube, where the image of the rings can be examined on a screen by aid of a microscope.

On warming up the vessel containing the prism under examination and its support, the rings at first appear very rapidly, and the black centres which appear are counted. After about three-quarters of an hour the equilibrium of temperature becomes established and the rings no longer pass, and the temperature is noted. Each passage of a black centre corresponds to a difference in elongation of half a wave-length between the prism and its support. The dilatation of the glass in question is then found by the equation—

$$\delta = \Delta \pm \frac{1472 f}{\theta} \times 10^{-8},$$

when Δ = Dilatation of the support;

f. The number of rings which pass;

θ . Difference between the extremes of temperature.

There may be a systematic error due to the graduation of the support, but this is reduced to a minimum by taking the mean of several experiments.

Most of the samples of glass used in this research were made in a Schloßing furnace in a platinum crucible, then cast into triangular prisms 30 to 40 m.m. long and of 8 to 12 m.m. width of face, in moulds of clay or wood charcoal; they were then annealed at a dull red-heat, and allowed to cool very slowly; when cool they were ground, and one end was polished. By using platinum crucibles there is no fear of any variation in the composition of the glass.

Our first endeavours were to try and verify the truth of the law connecting dilatation and chemical composition, which was admitted by Schott without sufficiently conclusive proofs; he thought that the dilatation of glass in

* Abridged from the *Bull. de la Soc. de l'Encouragement*, Series 5, Vol. ii., No. 6, June, 1897.

creased in a similar manner to density, by the simple addition of certain bodies. To decide this point we had to have recourse not to ordinary commercial glass of complex character, but to glass of very simple character prepared in the laboratory, and of which the constituent parts varied in proportions as widely apart as possible. After becoming convinced of the inexactitude of this law, we tried to find some precise qualitative indications of the manner in which variations in a given glass affected its dilatation.

The curves made for the comparison of the results give to dilatation a function of the volume, and that seems more reasonable than to imagine a relation between dilatation and the weight of the component parts of the glass.

Silicates of soda and potash are vitreous, from the state of NaO, SiO_2 and KO, SiO_2 up to SiO_2 ; but when more alkaline than $\text{NaO}, 2\text{SiO}_2$ and $\text{KO}, 3\text{SiO}_2$, they become too hygrometric to allow of practical measurement of the dilatation.

All the silicates of lithium we prepared became devitrified. Contrary to what occurs with alkaline silicates, the dilatation of silicate of lithium increases with the proportion of silica: it is true that the mixtures are not vitreous, so this increased dilatation is probably due to the presence of crystals of silica; the mean dilatation of quartz is, in fact, very high, viz., 1206×10^{-6} .

Borates of soda and lithia can be obtained in the vitreous state, from the condition of $\text{NaO}, 2\text{BO}_3$, and $\text{LiO}, 3\text{BO}_3$, up to BO_3 .

The only borate of zinc obtainable is $\text{ZnO}, 0.67\text{BO}_3$, and this becomes devitrified very rapidly into cubic crystals.

One per cent of boric acid is lost when heated to bright red for fifteen minutes, but this small loss does not interfere with the measurements of the easily fusible borates. The measurements made on a large number of samples of borates show that there is a very sharply defined minimum,—that the dilatation decreases with a slight increase in the proportion of the bases; it reaches the minimum, and then increases rapidly. This is an important fact, and shows by itself that Schott's law is incorrect; moreover, the same was observed with more complex samples of glass containing boric acid.

As much as 60 per cent of boric acid can be added to white glass, but beyond that the excess separates out, and even with this high proportion the glass is not really homogeneous. Bottle glass devitrifies when there is more than 15 per cent of boric acid present, but even under the microscope there is no trace of crystallisation.

From the results shown in the table it can be seen that the coefficient of dilatation of boric acid is about 350, and it is this figure that must be taken in considering the influence of small additions of boric acid in commercial glasses.

In certain cases oxide of lead is, like boric acid, capable of giving a minimum of dilatation; but the effect produced is never strongly marked, and does not appear to be general. By adding 6 per cent of oxide of lead, M. Damour increased the dilatation from 514 to 551. It can be added up to 60 per cent without devitrification in the case of bottle glass, but the glass is then no longer homogeneous.

Alumina, when in large proportion, has the effect of making the glass almost infusible: this necessarily limits its use. Oxide of lead seems to be the body which best dissolves alumina. The measurements made show that when increasing quantities of alumina are added a minimum of dilatation is reached, in the same way as with borate of lead. An interesting fact noticed is, that the separation of boric acid from $\text{PbO}_3, 3\text{BO}_3$, and from $3\text{ZnO}, 2\text{BO}_3$, is stopped by alumina, although when in the crucible these borates are as liquid as water. Finally, the addition of small quantities of alumina to glass has the effect of lowering the coefficient of dilatation; this effect is more marked the more acid the glass is.

Some of the conclusions to be drawn from this research are, that a large number of bodies, such as $\text{BO}_3, \text{PbO}, \text{CaO}, \text{MnO}, \text{Al}_2\text{O}_3$, &c., when added to the glass in small quantity, lower the dilatation first to a minimum, and afterwards increase it as the proportion added is raised. This has not been observed in the case of potash, soda, or silica glass; possibly it occurs with such a minute addition as not to be practicable. Nevertheless the double silicates of potash and soda have a lower dilatation than the corresponding simple silicates. Alumina, while greatly lowering the dilatation, allows us, on account of the special fixity it communicates to glass, to obtain very alkaline silicates of high dilatation, offering great resistance to water.

Fluoride of calcium, in spite of the high dilatation given by fluorine, only increases it very slightly, and when the proportion added becomes at all large the glass is completely devitrified. To sum up, the bodies studied can be divided into two classes:—1. Those which increase the dilatation, $\text{KO}, \text{NaO}, \text{LiO}, \text{CaO}, \text{P}_2\text{O}_5, 3\text{CaO}, \text{CaF}_2$, and cryolite. 2. Those which decrease the dilatation, $\text{BO}_3, \text{SiO}_2, \text{Al}_2\text{O}_3, \text{PbO}, \text{SiO}, \text{ZnO}, \text{Fe}_2\text{O}_3$, and the colouring oxides.

NOTE ON THE CHEMICAL COMPOSITION OF THE MINERAL RUTILE.

By B. HASSELBERG.

In my researches on the arc-spectrum of titanium I employed, as elsewhere stated (*Svenska Vetensk. Akad. Handl.*; also *Ap. F.*, v., 194—198, 1897), instead of the commercial metallic powder, a Norwegian specimen of the mineral rutile, mainly on account of the far greater steadiness of the arc thus formed. According to the hitherto published chemical analyses of this mineral (Dana, "Descriptive Mineralogy," fifth edition, New York, 1883, p. 160), I had no good reason to expect any foreign lines of importance other than those of iron, the more conspicuous of which would be present in any case on account of impurities in the carbons. However, upon examining the arc-spectrum of vanadium obtained from a specimen of this metal presented to Baron Nordenskiöld by Moissan, of Paris, I found, to my great surprise, that several of its strongest lines coincided exactly with faint lines in my titanium spectrum, thus indicating a very appreciable percentage of vanadium in the rutile analysed. This induced me to investigate more closely the spectra of other specimens of the mineral in question, particularly as I had the opportunity to select from among the rich collections of the Royal Mineral Cabinet varieties from different quarters of the world.

In the comparisons I have used only the part of the spectrum included between $\lambda 460$ and $\lambda 427$. This is sufficient, for in this region there is situated one of the most prominent groups of the whole vanadium spectrum, namely, the group $\lambda 441-438$, the presence of which in the spectrum of any rutile, even though feeble in intensity, would indicate indubitably the presence of a sensible percentage of the metal. In order to decide definitely concerning the coincidences, the above-named part of the vanadium spectrum was photographed upon the same plate with the same region of the spectra of the different rutiles, and on these plates the intensities of the rutile lines corresponding to vanadium were estimated on a scale in which 1 denotes the faintest, and 6 the strongest lines. A+ or — after a number indicates the intensity of the line in question to be nearer to this number than to the next. Thus, 1+ denotes an intensity greater than 1, but not attaining 1.2 and so on. In this way the following table has been constructed, which contains the results of the investigation of twelve rutiles, namely:—

Norway	1. Rutile from Kragerøe.
	2. " " Langøe.
	3. " " Lofthagen.

TABLE A.
Rutile from—

Vanadium		Kragerøe,	Langøe,	Loftes- hagen.	Käring- bricka.	Tachowaja.	Miask.	Binnen- thal	Yrieix.	Frei- berg.	Castilia.	Graves Mountain.	Arkan- sas.	Remarks.
λ	I													
4268.85	3	..	I+	..	I	I	I-	..	trace	I-	I	I+	trace	The word
71.80	3	..	..	..	I	..	..	..	..	..	..	..	..	'trace' in-
4330.15	3	..	I-	trace	I	trace	trace	..	..	trace	trace	I	..	dicates an
33.00	3	..	I	I	I	I	trace	..	..	trace	I-	I'2	..	intensity
41.15	3	..	I+	I	I-	I	trace	..	trace	I-	I	I'2	trace	too feeble
53.05	3.4	I	I+	I+	I'2	I+	trace	..	trace	I-	I	I'2	trace	to be es-
79.42	4.5	2	3	2.3	3-	2.3	2-	trace	2	2	2.3	2.3	2-	timated.
84.95	4.5	2	2.3	2+	2.3	2+	I'2	trace	2-	2-	2+	2+	I'2	
90.15	4.5	2	2	2	2+	2	I	..	I'2	I'2	2-	2	I+	
95.40	4.5	I	I'2	I'2	2	I'2	trace	..	I+	I	I+	2-	I	
4400.75	4	I'2	2-	I'2	2	2-	I	I+	I'2	I'2	2-	2-	I+	
06.85	4.5	..	I'2	I'2	2-	I'2	trace	..	I	I	I+	2-	I	
07.90	4.5	I'2	2-	2-	2	2-	I	trace	I'2	I'2	2-	2+	I+	
08.40	4	I'2	2-	2-	2	2-	I	..	I'2	I'2	2-	2+	I+	
08.65	4.5	I'2	2-	2-	2	2-	I	..	I'2	I'2	2-	2+	I+	
16.65	3	2	..	..	2	..	..	..	..	..	..	..	..	
38.03	3.4	..	I	I-	I	I	I	..	..	trace	trace	I	..	
41.90	3.4	I'2	2-	2-	2-	I'2	I'2	I+	..	I'2	I'2	2-	I	Ti
44.40	3.4	..	2-	2-	I'2	I'2	I'2	..	..	I'2	I'2	2-	I	Ti
52.12	4	..	I	I-	..	I-	trace	trace	..	trace	I-	I'2	..	
59.95	4	trace	I+	I+	I'2	I'2	I-	..	I	I	I	I+	I	
60.45	4.5	I	I'2	I'2	2-	2	I	..	I+	I'2	I'2	2-	I-	
62.55	3.4	trace	..	..	I	I	..	..	trace	trace	..	I	I	
69.90	3.4	..	..	..	I-	I-	trace	..	trace	trace	..	I-	I	
4545.62	3.4	..	..	..	I'2	I+	I	..	I	I	..	I+	I+	
49.85	3	3.4	..	..	3.4	3.4	3.4	..	3.4	3	..	3.4	3.4	Belongs
77.40	4	trace	..	..	I+	I+	I-	..	trace	I	..	I	trace	to Ti,
80.55	4	I	..	..	I'2	I'2	I	..	I	I+	..	I+	I	Co.
86.55	4.5	I	..	..	I'2	I'2	I-	..	I	I+	..	I+	I	
94.30	4.5	I	..	..	I'2	2-	I-	..	I	I+	..	I+	I	

TABLE B.

Chromium.													
4254.49	6	trace	3	3	2.3	3	trace	..	2.3	2.3	2.3	2.3	..
74.91	6	..	2.3	2.3	2-	2+	..	..	2	2	2	2+	..
89.87	6	..	2	2	2-	2	..	..	I'2	2-	2-	2	..

Sweden	4.	Rutile from	Käringbricka.
Russia	5.	" "	Tachowaja, Ural.
	6.	" "	Miask, Orenberg.
Switzerland ..	7.	" "	Binnenthal, Wallis.
France	8.	" "	Yrieix.
Germany	9.	" "	Freiberg.
Spain	10.	" "	New Castilia.
America	11.	" "	Graves Mountain, Lin-
	12.	" "	coln Co.
		" "	Magnet Caves, Arkansas

From Table A it will be seen that, with one exception, all the rutiles examined contain vanadium in varying proportions. This exception is found in the Anatas from Binnenthal, Canton Wallis, in Switzerland, in the spectrum of which the vanadium lines are almost absolutely wanting. This statement is not invalidated by the greater intensity of the two lines 4444.40 and 4441.90, for these lines belong without doubt to titanium, although they differ so very little in position from the vanadium lines that a separation on my spectrograms is impossible.

On comparing the intensities of the vanadium lines in the different specimens of rutile, the singular fact at once manifests itself that varieties from neighbouring lodes contain a very different percentage of the metal. Thus, among the Norwegian rutiles, the two specimens from Langøe and Lofteshagen contain vanadium in a much larger proportion than the Kragerøe rutile, and the same holds good for the two Russian and also for the American rutiles. This peculiarity finds a counterpart in the case of another component of some rutiles, namely, chromium, of which metal a very notable amount was discovered in the Swedish specimen from Käringbricka as early as 1803

(Dana, "Mineralogy," p. 161), and is now detected in some of the varieties under discussion.

In order to prove that the observed titanium lines are not to be ascribed to an impurity of the carbons, the spectrum of the latter was photographed with that of vanadium before introducing rutile into the arc. Besides the ordinary carbon bands the resulting plates show feebly only a few of the most conspicuous iron and calcium lines, but of vanadium not the least trace is seen. The purity of the carbons analysed is thus to be considered as entirely satisfactory.

While the presence of vanadium in the rutile thus forms a hitherto entirely unknown feature of this mineral, the presence of chromium in the Swedish variety was, as above stated, detected by chemical analysis as early as 1803, and has since then been verified in some other specimens. The present method of research, however, permits of a much easier decision in this respect on account of the occurrence of one of the strongest groups of the whole spectrum of chromium, viz., λ 4289.9, 4274.9, 4254.5 just within the part here photographed. It is not very difficult to find them out among the crowd of titanium lines on the photographs, and from their estimated intensities, to form at least an approximate idea of the greater or less quantity of chromium contained in the specimens examined. The results of these comparisons are given in Table B.

It will be seen that in different rutiles the chromium lines show differences of intensity, fully justifying the conclusion of a corresponding disparity in the amounts of the metal. Thus, while the Anatas and also the Arkansas rutile are absolutely free from chromium, and in the rutiles from Kragerøe and Miask only a feeble trace is

present, the other specimens contain a very considerable percentage of this metal. But the most peculiar feature in this respect appears upon comparing chromium with vanadium. It is thus found that in those varieties of rutile which contain vanadium in any very appreciable amount, chromium is also present, while a small percentage of the former metal is accompanied by a corresponding scarcity or even complete absence of the latter.

In conclusion, it should be remarked that in the case of the Norwegian rutile from Langöe the preceding results have been completely confirmed by ordinary chemical analysis kindly undertaken by Baron Nordenskiöld. It is thus evident that the accepted chemical analyses of the present mineral by no means possesses the completeness or accuracy which the usual chemical methods are capable of giving.—*Astrophysical Journal*, vi., No. 1, June, 1897.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING JULY 31ST, 1897.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolis Water Act, 1871.

London, August 10th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 189 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from July 1st to July 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in a previous report.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 189 samples examined all were found to be clear, bright, and well filtered.

The rainfall at Oxford during July was 2'57 inches, of which 2'25 inches fell on the 19th, 20th, and 21st inst.; the average for the last 30 years is 2'63 inches; this leaves a deficiency of 0'06 inch on the month. There was an excess of 0'06 inch on the first six months of the year; so this is exactly balanced by the deficit of this month; and the actual fall this year, up to the end of July, is identical with the 30 years' average, viz., 13'94 inches.

The results of our bacteriological examinations of 265 samples are recorded in the following table; we have also examined 53 other samples taken at special points, stand-pipes, wells, &c., making a total of 318 samples:—

	Microbes per c.c.
Thames water, unfiltered (mean of 27 samples)	497 ¹
Thames water, from the clear water wells of five Thames-derived supplies (mean of 133 samples)	83
Ditto ditto highest	496
Ditto ditto lowest	4
New River, unfiltered (mean of 27 samples) ..	754
New River, filtered (mean of 25 samples) ..	44
River Lea, unfiltered (mean of 27 samples) ..	2494
River Lea, from the clear water well of the East London Water Company (mean of 26 samples)	125

The average bacteriological quality of the London waters has been good during the month. On some days, however, in the case of two of the Thames-derived waters, there has been an excess of microbes, and the water from the Lea has not all the time been up to its best standard. We have been in communication with the Companies respecting certain alterations in the filter beds, which are likely to improve their efficiency. Our suggestions are being considered, and during the latter part of the month the bacterial quality of the water has greatly improved.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.
JAMES DEWAR.

PAINT TESTS.

MAX TOLTZ, in a paper read before the Civil Engineers' Society of St. Paul, December 7th, 1896, and printed in the *Journal of the Association of Engineering Societies* (June, 1897), has gone comprehensively into the comparative value of paints for protecting iron surfaces. The paints experimented with were (1) true asphaltic varnish paints; (2) so-called asphaltic varnishes, of inferior qualities; (3) black carbon paints, of which the vehicle is practically a varnish; (4) iron oxide paints; (5) graphite and silica graphite paints. Red-lead was not tested. Within the last ten years this material has been to a large extent discarded by progressive engineers, and although it has still warm advocates, even they are beginning to add carbon-black or graphite to it, says Mr. Toltz. One set of tests made by Mr. Toltz consisted in painting sheet-iron dishes, 12 inches diameter by 0'5 in. deep. The scale or skin was carefully removed before painting. Two dishes were then painted with each kind of paint, one receiving one coat and the other two coats, the first coat having dried at least a week before the second was put on. After the second coat had dried thoroughly, a given amount of water was placed in the dishes and allowed to evaporate at the ordinary temperature of the room, this being repeated until the dishes showed more or less rust. After most of the water had evaporated there remained at the junction around the edge a thin film of water, which in contact with the air, and the carbonic and other acids of the air, acted on the paint in such a way that the iron under it began to rust. In actual practice the same thing will happen, the only difference being that the rust will extend under the paint and will not show as plainly as on the dish. This is a severe test, but in Mr. Toltz's opinion no paint which fails to withstand it is desirable for the protection of iron and steel structures. The cheap asphaltum paints and iron oxide paints failed under this test. Another severe test consisted in exposing sheet iron coated with various kinds of paint to a temperature of 220–300° F., this test being of value as showing promptly whether a paint will keep its elasticity or will become so brittle that it may be easily removed from the surface.

In the discussion which followed this paper exception was taken to various statements made by Mr. Toltz, but the expressions of opinion were generally in support of his conclusions.

From our present knowledge, the following system for painting iron and steel bridges, and other metallic structures, is recommended by Mr. Toltz:—

First. Give the iron and steel a coat of the best grade of refined linseed oil, properly boiled and settled clear; or, still better, mix linseed oil with about 10 per cent of a good grade of lamp-black,—this coat to be applied at the mills, the iron or steel being first carefully cleaned from loose scales.

Second. After the structures have been erected, give

them one coat of real asphaltic varnish paint, made from the best grade of asphalt, linseed oil, and gum, compounded properly, so as to form a true varnish; or of a paint made from carbon black and properly boiled varnish, compounded of the best grade of linseed oil and gum. This coat should be carefully applied by a skilful painter, after the metal has been thoroughly cleaned from all loose scale, rust, shavings, filings, shrivelled oil or paint, grease, dirt, or any foreign matter, because it is of the utmost importance that the paint should be spread and worked in such a way so as to cover the surface properly, and be as free as possible from air-bubbles and form a continuous coating. This priming or first coat should be applied fairly thick, the thickness depending, to some extent, on the nature of the paint used. Before the second coat is applied, the first one should be thoroughly dried and hardened by natural oxidation, which will require at least ten days. If practicable, it would be a great deal better, as well as more economical, to apply the second coat not less than four weeks after the first one.

Third. As a second coat, a good grade of graphite paint is to be applied as thickly as possible, working the paint thoroughly with the brush. From the examinations made of the various grades of graphite paints, as far as graphitic pigments are concerned, there appears to be but little difference between them, provided, of course, that the pigment contains at least 33 per cent of pure graphite, the rest of the pigment being natural rock, ground very fine in pure linseed oil. The graphite paint should be bought in paste form, well ground, and contain not less than 70 per cent of pigment and 30 per cent, by weight, of the best quality of boiled linseed oil; the paste should be mixed with boiled linseed oil at the place where it is to be applied. No turpentine, no benzine, and no Japan or driers should, under any circumstances, be allowed in this paint.

Fourth. There are certain parts of steel or iron bridges, viaducts, or tunnels that should have an additional (third) coat of paint. These include such places, or parts of structures, as are directly exposed to the steam, fumes, and gases from passing engines. For such a coat some cheaper asphalt paints, applied very thickly over the coats above recommended, would be all-sufficient. Such a coat would protect the underlying primary coats for many years, preserving their natural toughness and elasticity, and preventing atmospheric action on the structure.

From the investigations made, as well as from practical experiments, it appears that the iron-oxide paints are not very desirable, at least for the first coat or two, for iron or steel; but as a third coat, for the protection of the underlying paints, they may be recommended.

However, the extensive investigation of the graphite paints that can be obtained in the markets to-day shows that, if properly applied, they are far superior to iron-oxide paints for the second or third coat, especially as they withstand the action of moisture and water much better than the best iron-oxide paint so far examined. Besides, a graphite paint, in paste form, well ground and mixed with boiled linseed oil, will not cost very much more per gallon than the cheapest iron-oxide paint in the market.

In recommending asphalt varnish paint or carbon paint for the first coat, great stress is laid upon the necessity of having the surfaces of iron or steel as free from moisture as possible while the structures are being painted, otherwise there is great danger that the coating will not adhere very firmly, and that it will thus actually nullify the value of the paint. This precaution is less important when an ordinary iron-oxide paint or red-lead paint, simply mixed with linseed oil, is used; because linseed oil itself has the property of absorbing moisture quite readily, whereas carbon or asphalt paint will not. The lack of this property in the two last-named paints is one of the principal reasons why they are superior to any other class of paints.

—Engineering and Mining Journal.

NOTICES OF BOOKS.

Twentieth Annual Report of the Connecticut Agricultural Experiment Station for 1896. Pp. 414. New Haven: The Tuttle, Morehouse, and Taylor Press. 1897.

THE Report on Food Stuff, which comes first in this volume, contains the results of examinations of 849 articles of food, of thirteen different kinds. With the exception of Martius yellow, found in minute quantity in certain samples of mustard, no poisonous adulterants have been found, though 254 samples were found to be adulterated and 24 were reported as doubtful. The most frequent adulteration was found in coffee. White strained honey was, as a rule, the purest article examined.

The law with regard to adulteration seems to be in a very lax condition. It appears that a "Dairy Commissioner" was appointed under the Statute regulating the sale of *imitation butter, molasses, and vinegar*, but no one is charged with the execution of the laws regarding the adulteration of milk, candy, spirituous liquors, drugs, and medicines. Boards of Health, we are informed, are permitted to act under the Statute regarding the adulteration of food; but there is no record of any action having been taken under any of these Statutes. Surely the work of the "Dairy Commissioner" is somewhat anomalous.

Under the Report on Commercial Fertilisers we note that 255 distinct brands of fertilisers have been offered for sale by fifty-two firms. During the year 492 samples of fertilisers and manurial waste products have been analysed, results of the examinations being given in detail; after which comes a comprehensive review of the fertiliser market. The average monthly quotations show that nitrate of soda has ruled lower this year than for some years previously, and there has been but a slight demand for sulphate of ammonia in the Connecticut market. Nitrogen in the form of animal matter has also been cheaper.

A number of experiments have been carried out, and are here described, on various agricultural matters, such as the prevention of potato-scab, and the susceptibility of various roots to potato-scab, and it was found from trials made on eight different kinds of roots—viz., radishes, parsnips, salsify, carrots, turnips (two kinds), mangolds, and beets—that the radishes and carrots alone remained unquestionably free from scab, the salsify and parsnips showed little if any, while the turnips gave averages of 21 per cent and 15 per cent of scab, the mangolds 40 per cent, and the beets 63 per cent.

Other experiments dealt with a leaf blight of melons, a destructive fungus disease of tobacco, the so-called "shelling" of grapes, &c.

The work done at the Experimental Station is of varied character and of considerable interest, while great care is evidently exercised in carrying out the plan of campaign. The volume closes with a good Index of fifteen pages.

Contribution to the Polyhedric Origin of Species. ("Contribution à l'Origine Polyédrique des Espèces"). By ARTHUR SORIA ET MATA. Part I., pp. 203. Madrid: Chamartin de la Rosa. 1897.

THE author has studied the regular polyhedra, from the point of view of making them, and combinations of them, represent the elements and their compounds; and from the curious figures evolved he has endeavoured, with a certain amount of success, to reproduce the structure of chemical compounds and minerals, showing the angles, faces, planes of cleavage, &c., and he claims that if, from a mass of polyhedral groups, as shown by the figures he has built up, he can show a regular and systematic arrangement which reproduces a large number of—if not

all—the known crystalline forms, it must well be admitted that his hypothesis holds good until another and more perfect one comes to replace it.

Starting from the idea of an atom, which idea was not conceived without many doubts and much hesitation, we must admit motion, or no phenomena could take place; and from this point he works up to lines of atoms moving in unison and communicating their movement one to another, the total force being neither augmented or diminished, though passing and changing without ceasing from lines to faces, angles, and solid figures; this is not a long step, and he classifies all the figures necessary for his hypothesis into three groups, viz.:—the tetrahedron, the common origin of all bodies of three dimensions; the cube, which contains the beta-tetrahedron and the octahedron, the typical form of chemical species and minerals; and the dodecahedron, which contains many regular polyhedra, and represents the probable initial form of the cell, and all the animal and vegetable species. The theory is a pretty one, but is open to many objections,—still, as the author says, it may stand till another comes to replace it.

We cannot agree with the author's supposition of the interpenetrability of atoms; this supposition must concede mass, and therefore if two "masses" can penetrate each other "until their centres are coincident, without losing their respective independence of movement," there is an end of matter, as—carrying the argument further—all atoms would be self-contained in the mass, position, or volume, whichever it may be, of one,—which is absurd.

The book is accompanied by four large sheets of thick paper, on which are marked out a number of figures, all ready for cutting out and glueing up into the forms referred to in the letterpress.

OBITUARY.

VICTOR MEYER.

THE sudden and unexpected death of Professor Dr. Victor Meyer, on the 7th inst., has left a painful impression not merely at Heidelberg, but throughout the chemical world. The regretted event was at first ascribed to apoplexy, but it has since been stated—on what evidence it is not certainly known—that Prof. Meyer fell a victim to poisoning. We should add that those who regard the death of Prof. Meyer as suicide, allege that he suffered from domestic troubles.

Victor Meyer, the son of the calico-printer, Jacques Meyer, of Berlin, was born on September 8th, 1848. After his earlier education at the Werder Gymnasium he entered, in his sixteenth year, the University of Berlin, and after one term that of Heidelberg, where he devoted himself to the study of Chemistry under Prof. Bunsen, whose assistant he became. After graduating at Heidelberg he continued his studies at Berlin under A. von Baeyer. In 1871 he was called to the Polytechnicum at Stuttgart as First Assistant of Fehling, and (titular) Professor of Organic and Theoretical Chemistry. After a year he received a call as Professor of General Chemistry at the Polytechnicum at Zurich, *vice* Wislicenus. In 1885 removed to Göttingen, and in 1889 he was transferred to the University of Heidelberg as the successor of Bunsen, who had recommended him as the highly gifted of his pupils. Under his leadership chemical studies at Heidelberg experienced a rapid rise. In the last year from fifty to sixty applications had to be rejected in every term.

Through Victor Meyer's researches our science experienced important extensions in various directions. The methods of determining vapour densities were greatly simplified and facilitated by his procedures. His results at high temperatures (up to 1800° and beyond) yielded a firm basis for pyro-chemistry. We are indebted to him for

the discovery of the aldoximes and ketoximes (classes of bodies which become of fundamental importance for the recognition of the aldehyds and ketones). They became of prominent importance in the development of stereochemistry, especially that of nitrogen.

His most brilliant discovery in organic chemistry was that of thiophene in benzene, proceeding from which Victor Meyer may be said to have created a new branch of chemistry. As a Professor he was unsurpassed; few Academic teachers equalled him in the art of stimulating his hearers. Both his discourse and his carefully prepared experiments afforded his pupils a high enjoyment.

In 1885 Prof. Victor Meyer was elected an honorary member of the British Chemical Society.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv. No. 4, July 26, 1897.

Composition of Drainage Waters.—P. P. Dehérain. —From March, 1895, to March, 1896, the fields kept in fallow alone have yielded drainage water; the lands sown with annual plants, already impoverished in water by the vegetation itself, have been so thoroughly dried by the exceptional temperature of the autumn of 1895 that the autumnal rains could not saturate them. If we calculate for the surface of a hectare, we find that the drainage of the plots in fallow has removed 109 kilos. of nitric nitrogen—a figure analogous to that of 1893 and 1894, but very inferior to that of 1892. The rain has been very unequally distributed during the agricultural year, March, 1896, to March, 1897. Scanty at the outset, it became abundant in June, moderate in July and August, but it was extremely plentiful in September and October (130 and 136 m.m. respectively). The quantities of nitric nitrogen in the fallow land, deprived of nitrogenous manures, rose, during the moist years, to 200 kilos. per hectare, representing 1250 kilos. nitrate of soda, and exceed the requirements of the most greedy crops. The soils under crops elaborate a much smaller quantity of nitrates, for the abundant evaporation of the herbaceous plants dries the soil so completely as to admit of an energetic nitrification. When rain is very abundant we obtain, without nitrogenous manures, very good harvests, containing a sufficiency of nitric nitrogen.

Researches on the Conditions in which there occur Elements other than Carbon in Cast-Irons and Steels.—Ad. Carnot and M. Goutal.—This paper will be inserted in full.

Explanation of a Phenomenon attributed to a Magnetic Deviation of the X Rays.—Sir G. G. Stokes. —Already inserted.

Phthalic Green: its Preparation and Constitution.—A. Haller and A. Guyot.—Otto Fischer has given this name to a green pigment obtained in small quantities by causing phthalyl chloride to act upon dimethyl. The authors have found that in addition to diphenylphthalide there are formed small quantities of a new compound, $C_{20}H_{18}O$. This compound is formed by other methods, and in particular by the action of aluminium chloride upon phthalyl and benzene tetrachloride in the diphenylanthrone described by one of us in a former communication. The compounds here described are the hydrochlorate, $C_{32}H_{35}N_3OCl + H_2O$; the nitrate, $C_{32}H_{34}N_3O.NO_3$, is a very stable substance. The chloroplatinate, $(C_{32}H_{34}H_3OCl + 3HCl)_3PtCl_4$, is obtained in fine vermilion-red crystals. The leucobase of phthalic green has the composition $C_{32}H_{35}N_3O$.

Transformation of the X Rays by Metals.—G. Sagnac.—The various metals exert an elective absorption upon the X rays. At the same time the superficial layer of the metal emits anew rays much less easily transmitted than the X rays by mica, aluminium, black paper, and the air itself. These new rays are themselves transformed by aluminium. We foresee that we shall gradually succeed in filling up the space which separates the X rays from the known ultra-violet rays, and perhaps identify them with such rays.

Photographic Veiling in Radiography.—P. Villard.—Radiographic proofs often present a veiled aspect, especially in the case of thick objects. We admit readily that this result is due to X rays of a peculiar nature capable of traversing almost all bodies without notable absorption.

Action of X Rays upon the Temperature of Animals.—L. Lecerclé.—Already inserted.

Line Spectrum of Carbon in Fused Salts.—A. de Gramont.—Tables of the wave-lengths forming the line-spectrum of carbon as recognised and measured in melted carbonates.

Relation between the Polymerisation of Liquids and their Dissociating Power upon Electrolytes.—Paul Dutoit and Mlle. E. Aston.

A New Group of Amidines.—Fernand Muttolet.—These two papers will be inserted as soon as possible.

Procedure for Determining Acetylene applicable to Carbides of the Form $R-C\equiv H$.—M. Chavestelon.—The author brings back the determination of acetylene to an acidimetric estimation.

Determination of Lime, Alumina, and Iron in Mineral Phosphates.—L. Lindet.—This memoir will be inserted in full.

Absorption of Oxygen in the Fracture of Wines.—J. Laborde.

Bacteriological Study of Ambergis.—H. Beauregard.—Already inserted.

Revue Générale des Sciences Pures et Appliquées.
No. 14, July 30, 1897.

Annual Review of Pure Chemistry.—A. Etard.—One of the most important events of the year in pure chemistry is the liquefaction of fluorine by Messrs. Moissan and Dewar, whose paper has already been printed in full in these columns. Of the other less important work, the author sees nothing but a little more confusion, owing to the monotonous continuity of scientific productions of unequal value. The science of chemistry is extending every year in new directions. It now includes pressure, cold, electricity, &c., &c.. It is being enriched principally by the determination of volumes, by the measurement of constants, and in more minute work striving to show if what we have so long admitted is really true. After a long period, during which it has seemed that science was getting so pure as to be useless, *savants* are coming back to the traditions of Gay-Lussac and Berthollet, and are endeavouring to adapt the modern lofty ideas to the wants of mankind. Thus, Lord Rayleigh proposes to make the direct oxidation of the nitrogen of the air commercially practicable by means of the electric currents which are now at the disposal of chemists. He found that with an electromotive force of 6000 volts the absorption of air in a 7-litre flask, in which played a spray of potash, reached 6880 c.c. per hour. Mr. Liversidge has tested various deposits of maritime origin for gold, and has found that the Stassfurt salts contain 0.13 grm. per ton. But it is not only gold that is so widely disseminated, but, as Mr. Hartley has shown, also the so-called rare earths and metals. In 92 different iron ores he found always either silver, rubidium, copper, gallium, indium, or thallium. Mr. Shenstone has found that the halogens Cl, Br, and I combine with mercury when in a

state of perfect dryness, but ozone will not do so except in the presence of aqueous vapour. Mr. Chattaway has studied the composition of iodide of nitrogen, and comes to the conclusion that NH_3I_2 is the most probable formula, certainly the rapport $N : I_2$ has been established.

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THE ARGENTAURUM PAPERS,

No. 1.

Some Remarks concerning Gravitation,

ADDRESSED TO

THE SMITHSONIAN INSTITUTE, THE ACADEMIE DES SCIENCES, THE ROYAL SOCIETY,
AND ALL OTHER LEARNED BODIES.

BY

STEPHEN H. EMMENS,

*Member of the American Institute of Mining Engineers, Member of the American Chemical Society,
Membre Fondateur of the Société Internationale des Electriciens, Sometime Fellow of the
Institute of Actuaries of Great Britain and Ireland, Member of the United
States Naval Institute, Member of the Military Service
Institution of the United States.*

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The above-mentioned work is Published by the Plain Citizen Publishing Company, 1, Broadway, New York City, N.Y., U.S.A.

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N.B. We widely advertised an offer of 10,000 dols. in Prizes to any persons who would point out any scientific errors in the above-mentioned book. NO CLAIMANT HAS COME FORWARD.

THE CHEMICAL NEWS.

VOL. LXXVI., No. 1971.

LABORATORY NOTES FROM NEW ZEALAND.

By WILLIAM SKEY,
Analyst to the Mines Department, N.Z.

1. If gold in potassic cyanide (weak or strong) is connected with platinum in an acid (say hydrochloric acid) and interpolar connection made, a stream of hydrogen is given off from the platinum. For this experiment it is necessary to have the negative pole very small as compared with the size of the gold. I do not get this reaction if the platinum is in an alkaline solution,—probably because there is an oxidation of the hydrogen by the free oxygen in solution, induced by the affinity of potash for water. Copper is easily precipitated upon a gold or silver pole out of its sulphate, by gold or silver in a cyanide solution.

2. If platinum or gold, in a solution of tannic acid and potash, is paired with platinum or gold in an acid such as acetic or sulphuric acid, an evolution of hydrogen also takes place, and continues till all the tannic acid is oxidised. By the use of the tannic acid or tannin battery of a few cells, we can get all the manifestations of a very intense current. To conserve the tannic acid there should be a layer of kerosene oil upon it, to keep out the air; or the cells may be closed with cork, and the connections run through it.

In these two experiments it appears that, in the one case, it is the hydrocyanic acid of the hydrocyanate of potash that is decomposed, because this compound must be in the nature of things the most easily decomposable electrolyte present under the circumstances. Nature's work is always done, as we know, upon the lines of *least resistance*. In the second experiment it appears that water is decomposed. In neither of these experiments is it at all likely that any assistance is given to the operation by the electric current generated. The metals and the electrolytes merely allow of electricity being made, the free flow of which is necessary or favourable to continuous chemical action.

3. Platina that has been for three days in a solution of potash is still positive in that solution to platina in acids generally, and throws down gold on platina from its chloride or potassic aurate, also silver from its ammoniate. The platina was the purest I could obtain, and was digested for a long time with nitric acid, then well washed before use. An ignition of the metal prior to use did not affect the results.

4. Platina in a concentrated solution of common salt alkaliised with potash that had been just boiled to expel air; also deposited gold from its chloride on a platina wire. As paired with platina in muriatic acid, it vigorously deflected a galvanometric needle for a short time; then deflected it feebly, but persistently, for an indefinite time.

The vigorous deflection may denote an oxidation of the platina itself, while the after deflection may denote an induced combination of the small minute quantity of oxygen and nitrogen that find their way to the surface of the platina that is in the saline solution. This has yet to be tested.

5. Platina in a weak or strong potash solution, paired with platina in a solution of ferrosulphate, is positive thereto, and if a decomposition cell with platina poles and an electrolyte of auric chloride be put in the circuit, a little gold is soon deposited upon that pole which is metallically connected with the platina in the potash solu-

tion. These results clearly show that a chemical action is taking place in the potash cell that is of a more intense character than that which we have in the case of oxidation of protoxide of iron to the sesquioxide.

For these kind of experiments bibulous paper for the electrical connection is not at all adapted, as the mixing of the electrolyte has to be avoided. The best method that I have yet devised is the "*gelatine connection*." A strong aqueous solution of gelatine is poured into a glass tube bent to a convenient form till it is full. This when cold is very convenient for the purpose, and absolutely prevents the blending of the solutions for days or weeks, according to their nature. To increase their conductive power where necessary, a little salt may be used. When made very stiff, even, it still conducts, showing that the molecules therein are still capable of moving to form the interpolar lines.

6. If an insulated voltaic cell be connected with the insulated silver plates—say 6 inches square and about an inch apart—in the air, a current of electricity still obtains; this is so weak, however, that a pretty sensitive galvanometer does not indicate it. By passing the current into a solution of auric chloride by means of platina poles gold is deposited in a day or so (on the positive pole) in a very thin, coherent, continuous, and bright film. With the positive pole of gold the action is much accelerated.

RESEARCHES ON THE STATE IN WHICH ELEMENTS OTHER THAN CARBON ARE FOUND IN CASTINGS OF STEELS.

By AD. CARNOT and M. GOUTAL.

We propose here continuing the study of the chemical condition of the elements which enter into the composition of castings and steels, occupying ourselves first with the metals properly called manganese, copper, nickel, and chromium; then the rarer elements generally ranged along with the metals titanium, tungsten, and molybdenum.

Manganese.—The experiments which we have already described show that manganese has an especial affinity for sulphur and silica, and that, when it is in small proportion in a casting, it may be found entirely in the state of manganese sulphide or silicide. When it is in a large proportion, the solvents which we employ for iron cause the manganese to disappear at the same time.

Copper.—Potassium cupro-chloride does not allow us to isolate the copper contained in a steel; but we may effect this easily by the use of a weak acid, such as hydrochloric acid at 5 per cent, if it is employed with the exclusion of air, *e.g.*, in a current of carbonic acid gas.

Nickel.—Nickel disappears entirely under the action of the neutral potassium cupro reagent.

Chromium.—Ferro-chromes of a high standard are not readily attacked by acids. Hence we have been obliged to operate upon steels containing not more than 2.50 per cent of chrome. The insoluble residues consist of chrome, iron, and carbon.

Titanium.—The ferro-titanates may be attacked either by acids or by the cupric salt. The titanium is uncombined.

Tungsten.—The attack of a tungsten steel by dilute hydrochloric acid, at a gentle heat and with the exclusion of air, leaves as residue a compound of iron and tungsten of the composition Fe_3W .

Molybdenum.—Molybdenum steels, treated with dilute acids in the absence of air, leave a residue answering exactly to the Fe_3Mo_2 .

In fine, manganese, nickel, copper, and titanium seem to be simply dissolved in the steels; a portion of manganese may be in the state of sulphide or silicide in the cast metals,

Chromium forms complex and perhaps multiple compounds with iron and carbon.

Tungsten and molybdenum are in the state of definite combinations with iron represented by the formulæ Fe_3W and Fe_3Mo_2 .

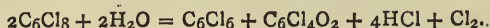
These elements, generally considered as metals, behave therefore in steel like non-metals, whilst arsenic, on the contrary, plays a part analogous to that of the true metals.—*Comptes Rendus*, cxxv., p. 221.

ON A COLORIMETRIC REACTION OF DISULPHURIC ACID.

By M. E. BARRAL.

WHEN the powdered parabichloride of hexachloridised benzene, $\text{C}_6\text{Cl}_6\text{Cl}_2^{1-4}$, is dissolved gradually in sulphuric acid containing disulphuric acid, a magnificent reddish violet colouration is produced.

By adding water or ordinary sulphuric acid, or even by leaving the mixture in contact with moist air, the colouration disappears as soon as all the disulphuric acid has been transformed into SO_4H_2 . Under the influence of water and SO_4H_2 , C_6Cl_8 gives both perchloridised benzene, perchlorised quinone, hydrochloric acid, and free chlorine:—



To show that this colouration is not due to the impurities of the Nordhausen acid, and that it is not produced in the presence of the anhydride, I prepared some sulphuric anhydride synthetically by passing $\text{SO}_2 + \text{O}$ over spongy platinum. By grinding together C_6Cl_8 and SO_3 , being careful to exclude all moisture, nothing is produced, while a beautiful reddish violet colouration appears as soon as the mixture is exposed to moist air.

Volumetric Estimation.—The disappearance of the colouration at the moment when all the disulphuric acid is transformed into SO_4H_2 , enables us to make use of $\text{C}_6\text{Cl}_6\text{Cl}_2^{1-4}$ as an indicator in the volumetric estimation of disulphuric acid.

A given volume of Nordhausen acid (25 c.c. for example) is poured into a flask, a little powdered C_6Cl_8 is added, and the mixture is shaken up until the colouration appears. We then run in, drop by drop, some carefully titrated sulphuric acid (acid at 66° , to which is added one-tenth its weight of water), until the disappearance of the reddish tint: a simple calculation enables us to estimate the quantity of disulphuric acid present in the Nordhausen acid.

This method of estimation, which enables us to act directly on the acid, gives excellent results in cases when the acid is colourless or only faintly coloured. Unfortunately it loses a good deal of its sensitiveness with commercial Nordhausen acids; these being as a rule more or less yellow, or even brown, the disappearance of the reddish tint is difficult to observe very accurately.—*Journ. de Pharm. et de Chim.*, Series 6, vol. vi., No. 3.

CONTRIBUTION TO THE STUDY OF THORIUM.

By G. URBAIN.

1. WHEN we wish to obtain thorium from thorite, we treat the latter with hydrochloric acid and evaporate to dryness to render the silica insoluble. We separate the lead and the tin by means of sulphuretted hydrogen, which is afterwards driven off by boiling. We then add a little chlorine-water to peroxidise the iron before precipitating it with oxalic acid, if the mineral does not contain the alkaline earths in any notable quantity. The oxalates are then calcined, and the oxides converted into

anhydrous sulphates, which are dissolved by projecting them—a little at a time—into iced water.

This is rather a delicate operation; above all, when working on large quantities, because the sulphates of the cerium group have a strong tendency to become hydrated, and for this reason are very difficult to dissolve. It is therefore necessary to take great care to rid these sulphates of all trace of free acid, and to add them to the water in minute quantity only if we wish to get them entirely dissolved. It has been noticed that a little acetate of ammonia increases the solubility of the sulphates of this group enormously.

A cold saturated solution of acetate of ammonia, diluted with twice its bulk of water, dissolves flocculent sulphate of thorium instantly. The double potassic sulphates of thorium, cerium, lanthanum, and didymium also dissolve with great ease in the same reagent, and they are not precipitated on boiling.

The sulphate of thorium containing $8\text{H}_2\text{O}$, when treated with a saturated solution of acetate of ammonia, forms a felty mass composed of fine needles of acetate of thorium. In a weaker solution it is completely dissolved. It is slightly precipitated on boiling, and at the same time acetic acid is given off, and the turbidity does not disappear by adding water or acetic acid, but only on the addition of hydrochloric acid. The presence of free acetic acid diminishes the solubility of the acetates very considerably.

Formate of ammonia appears to behave in a very different manner, and I intend to return to this subject later on.

The method of purifying thorium, as described by Nelson, is long and tedious. It consists of precipitating the thorium in the state of a flocculent sulphate, and it gives very bad results.

The solution of the oxalate in oxalate of ammonium carries with it a large proportion of cerium. The hypsulphite method gives results hardly better than the incomplete precipitations obtained by alkalis. None of these methods give even approximately pure thorium, except by repeating the operations several times.

2. I obtained thorium, which did not show a trace of cerium, very rapidly by the action of peroxide of hydrogen on its hydrate, with the formation of an acetylacetonate. For this purpose I used a relatively pure thorium which I had prepared in the following manner:—The raw oxalates were treated with oxalates of ammonia, and instead of precipitating the oxalate from this solution by means of an acid, I found it more advantageous to precipitate it by ammonia.

The acetylacetonate of thorium was obtained very easily by treating the hydrate of thorium, suspended in dilute alcohol, by acetylacetone, evaporating to dryness on the water-bath, and taking up with chloroform, which dissolves the acetylacetonate of thorium and leaves it, on slow evaporation, in well-defined crystals.

The molecular weight was taken in bromide of ethyl by the cryoscopic method.

Substance used	1'308 grms.
Weight of solvent	94'157 "
Loss of weight	0'26 "

	Found.	Calculated.
Molecular weight	630	628'4

This was done by taking the atomic weight found by Nelson, viz., 232'4.

I have also estimated the thorium by treating the acetylacetonate with nitric acid and then calcining the nitrate:—

Substance used	0'570 grm.
Thoria found	0'241 "

	Found.	Calculated.
Per cent.	42'2	42'1

Combustion gave me the following figures:—

Substance used	0.570 grm.
Water	0.115 "
Carbonic acid	0.389 "

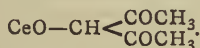
Summary of Analyses.

	Found.	Calculated.
Th.. .. .	37.09	36.98
C	37.75	38.19
H	4.54	4.45
O	4.54	20.38

Total.. .. 100.00

The acetylacetonate of thorium therefore corresponds to the formula $\text{Th}(\text{C}_5\text{H}_7\text{O}_2)_4$.

Under similar conditions, the hydrate of cerium is transformed into a grey amorphous body, almost insoluble in chloroform and alcohol. The analysis of this body shows it to be an ill-defined compound very like a basic salt—



We can obtain acetylacetonate of thorium just as easily by the double decomposition which takes place between acetylacetonate of soda and any salt of thorium. The liquid is well shaken up with chloroform, which is then distilled off.

3. Acetylacetonate of thorium, though but slightly soluble in water, is dissolved by most organic solvents. It is very soluble in alcohol and chloroform, but less so in ether and bromide of ethyl. Its crystals are of the clinorhombic system, and when formed from ether or chloroform they appear as elongated prisms:—

$$\begin{aligned} e' : e' &= 82^\circ 16' \\ e' : m &= 30^\circ 05' \\ m : m &= 80^\circ 55' \end{aligned}$$

When formed from bromide of ethyl the crystals are only microscopic.

Acetylacetonate of thorium melts at $171-172^\circ$ and sublimes *in vacuo*.—*Bull. Soc. Chim.*, Series 3, vol. xv., p. 347.

THE SEPARATION OF ALUMINUM AND BERYLLIUM BY THE ACTION OF HYDROCHLORIC ACID.*

By FRANK S. HAVENS.

In a former paper (Gooch and Havens, *Am. Journ. Sci.*, ii., December, 1896) a method was described for the determination of aluminum in the presence of iron, based upon the fact that the hydrous aluminum chloride, $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$, is practically insoluble in a mixture of strong hydrochloric acid and anhydrous ether saturated with hydrochloric acid gas, while the ferric chloride is entirely soluble in that medium.

The work to be described in this paper is an extension of this process to cover the separation of aluminum from beryllium, with the subsequent determination of the beryllium by weighing as the oxide after conversion to the nitrate and ignition.

The aluminum chloride solution was prepared by dissolving the so-called pure aluminum chloride of commerce in as little water as possible, precipitating, and washing free from iron with strong hydrochloric acid, dissolving the chloride thus obtained in water, precipitating the hydroxide by ammonia, washing the precipitate free from all alkalis, and re-dissolving it in hot hydrochloric acid. From this solution, after cooling, gaseous

hydrochloric acid precipitated the pure hydrous chloride. This prepared chloride was dissolved in water and the solution standardised by precipitating with ammonia the hydroxide from weighed portions and weighing as the oxide. The solution of beryllium used was made by dissolving in water beryllium chloride found to be free from iron by the sulphocyanate test, and giving no precipitate when tested by the gaseous hydrochloric acid process, to be described later on. This was standardised by precipitating with ammonia the hydroxide from weighed portions and weighing the ignited oxide in the usual manner.

In the experiments of Table I., weighed portions of the aluminum solution were mixed with portions of the beryllium chloride solution representing from 0.01 to 0.10 grm. of the oxide, an equal volume of a mixture of strong hydrochloric acid and ether (taken in equal parts) was added to the solution of the mixed chlorides, and the whole was completely saturated with gaseous hydrochloric acid while kept at a temperature of about 15°C . by immersing the receptacle in running water. Ether was added, equal in volume to the aqueous aluminum and beryllium solutions originally taken, and the current of gas again turned on until saturation was complete. By this treatment there is present at the end of the saturation a volume of ether equal to that of the aqueous hydrochloric acid introduced and generated. The finely-crystalline precipitate of aluminum chloride was caught on asbestos in a filter crucible, washed with a previously-prepared mixture of hydrochloric acid and ether in equal parts, saturated at 15°C . with hydrochloric acid gas, and dried for half an hour at a temperature of 150°C . It was next covered with a layer of pure mercuric oxide, which had been tested and found to leave no residue on volatilising, and the crucible was gently heated over a low flame under a ventilating hood and finally ignited over the blast.

TABLE I.

	Al_2O_3 taken in solution as the chloride.	Al_2O_3 found.	Final volume. C.m.s.	Error.
1.	0.1046	0.1044	12	0.0002—
2.	0.1046	0.1038	12	0.0008—
3.	0.1067	0.1066	12	0.0001—
4.	0.1071	0.1063	12	0.0008—
5.	0.1059	0.1054	30	0.0005—

From these results it is obvious that the aluminum chloride may be determined in the presence of beryllium chloride with reasonable accuracy.

The beryllium may be recovered in the filtrate from the aluminum chloride by precipitation with ammonia after nearly complete evaporation of the acid. It was found, however, upon trial that the conversion of the chloride to the oxide without precipitation and filtration may be easily accomplished by treatment with nitric acid and ignition. The results of Table II. indicate this clearly. In these experiments weighed portions of the beryllium solution were evaporated just to dryness on a radiator, care being taken not to heat to the volatilising-point of the beryllium chloride, a few drops of strong nitric acid were added, the liquid was evaporated, and the residue heated—at first gently, to break up the nitrate safely, and finally on the blast. It was found that this conversion of the beryllium to the nitrate can be carried on in platinum without attacking that metal appreciably, providing care be taken to remove thoroughly all excess of hydrochloric acid before the nitric acid is added to the dry residue.

TABLE II.

	BeO taken in solution as the chloride.	BeO found.	Error.
1.	0.0483	0.0481	0.0002—
2.	0.0483	0.0483	0.0000
3.	0.1076	0.1085	0.0009+

In Table III. (1 to 9) are given the results of experiments in which both the aluminum and the beryllium

* Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, Series 4, Vol. iv., No. 20, August, 1897.

were determined—the former by precipitation as the hydrous chloride and weighing as the oxide after igniting with mercuric oxide; the latter by the conversion of the chloride, through the nitrate, into the oxide. In Expt. 10 (made to get a comparison of the methods) the beryllium was recovered by precipitating the hydroxide with ammonia from the partially evaporated solution of the chloride after removing the aluminum.

In Experiments 1 to 5 inclusive, the aluminum was determined exactly as previously described; in 6 and 7, the solutions (being originally larger) were concentrated by evaporation previous to the addition of the ether and hydrochloric acid mixture. In Experiments 8, 9, and 10 the treatment was varied advantageously by saturating the aqueous solution directly with hydrochloric acid gas before adding an equal volume of ether, and completing the saturation.

TABLE III.

	Al ₂ O ₃ taken in solution as the chloride.	Al ₂ O ₃ .	Error.	C.m.s.	Final volume.	BeO taken in solution as the chloride.	BeO found.	Error.
1.	0.1059	0.1058	0.0001	—	12	0.0198	0.0204	0.0006+
2.	0.1053	0.1044	0.0009	—	12	0.0194	0.0196	0.0002+
3.	0.1065	0.1059	0.0006	—	12	0.0197	0.0205	0.0008+
4.	0.1068	0.1060	0.0008	—	12	0.0199	0.0207	0.0008+
5.	0.1049	0.1047	0.0002	—	12	0.0198	0.0208	0.0010+
6.	0.1060	0.1057	0.0003	—	12	0.0977	0.0969	0.0008—
7.	0.1064	0.1063	0.0001	—	12	0.1085	0.1084	0.0001—
8.	0.1046	0.1038	0.0008	—	30	0.1083	0.1087	0.0004+
9.	0.1051	0.1048	0.0003	—	30	0.1071	0.1078	0.0007+
10.	0.1076	0.1075	0.0001	—	30	0.1086	0.1094	0.0008+

These results are plainly very good.

The manipulation of the process is not difficult. The gaseous hydrochloric acid is most conveniently produced by the well-known method of treating with strong sulphuric acid, in regulated current, a mixture of strong aqueous hydrochloric acid and common salt. A platinum dish hung in an inverted bell-jar, provided with inlet and outlet tubes through which the current of water for cooling is passed, makes the best container for the solution to be saturated with the gas. It is advantageous to arrange the filtration upon asbestos, so that the filtrate and washings may be caught directly in the crucible (placed under the bell-jar of the filter-pump) in which the subsequent evaporation is to be effected. The heating of the strong acid solution must be gradual and conducted with care to prevent mechanical loss by a too violent evolution of the gaseous acid.

It only remains to thank Professor Gooch for kind suggestions and advice.

ON THE OCCURRENCE OF VANADIUM IN SCANDINAVIAN RUTILE.*

By B. HASSELBERG.

THE problem of assigning to each chemical element its definite emission spectrum has been carried perceptibly nearer to its solution, since the necessary basis was established by the classical work of Rowland. Yet when the attempt is made to extend it to the fainter radiations, as well as to the principal lines of the spectrum, the question becomes complicated to such an extent that an exhaustive solution seems to be well nigh hopeless. It is therefore necessary, in this department of spectroscopic

research, to be contented with a series of approximations; and no very great surprise need be felt if in a single case, where every effort has been made to eliminate such impurities as were most likely to occur, other impurities have been encountered, the presence of which there was in the beginning scarcely any reason to suspect. The present lines are devoted to a preliminary account of such a case as the above.

For producing the spectrum of titanium in the electric arc I have used titanous acid in the form of rutile with materially better success than when commercial titanium was employed, since the metal burns much too quickly when introduced into the arc, and is scattered in all directions. This rutile, which was kindly obtained for me by Baron Nordenskiöld, comes from Kragerø, in Norway. As in other kinds of rutile, its chief component is titanous acid, since, according to analysis of a number of varieties of this mineral (Dana's "Descriptive Mineralogy," 5th edition, 160, New York, 1883), the only other constituent to be expected is oxide of iron in the proportion of from 1 to 2 per cent. After eliminating the iron lines which are thus caused to appear on the spectrograms, as well as other metallic lines, whose presence was revealed by comparison with my own investigations of metallic spectra and with those of Kayser and Runge, I considered myself justified in ascribing the remaining lines to pure titanium, or at least in considering that only a few isolated cases remained in which contamination by a foreign metal might subsequently be proved. The continuation of my spectroscopic researches has shown, however, that this does not entirely hold good, since among the fainter and faintest lines of my titanium spectrum there are several that doubtless belong to vanadium. Having obtained through Baron Nordenskiöld a large piece of this metal, which was made by Moissan, of Paris, in the electric furnace, I recently began a re-examination of the spectrum, and discovered several strong groups of lines in the blue and violet parts, the approximate wave-lengths of which agreed very closely with those of faint lines previously measured in the spectrum of titanium. In order to obtain a final decision on this point, the parts of the spectrum in question of vanadium and rutile were photographed in juxtaposition on the same plate, in the usual manner, and compared line by line. The result of this investigation of approximate coincidences is shown in the accompanying table.

In the first two columns of this table are given the approximate wave-lengths and intensities of vanadium lines, in the region λ 403— λ 405, for which corresponding fine lines occur on the comparison plates of the spectrum of Norwegian rutile. These lines are given, with their intensities, in the two following columns, the wave-lengths being those of my former catalogue of titanium lines. As will be seen, the coincidences are almost all exact; and hence the corresponding faint titanium lines are to be removed from the titanium spectrum as properly belonging to vanadium.

I must confess that I have been greatly surprised by this result. It will be granted, I think, that there were no possible reasons for suspecting it in advance, since vanadium has never been found in any of the numerous varieties of rutile hitherto known. Under these circumstances it seemed to me desirable to subject another kind of rutile to the same test. For this purpose Swedish rutile from Käringbräcka in Westmanland was chosen, one reason among others for doing so being that the results of Ekeberg (*Svenska vetensk. Akad. Handl.*, xlvii., 1893; Dana's "Mineralogy"), according to which this particular kind also contains chromium, could be tested at the same time. A double exposure to the spectrum of this mineral and of vanadium in the region λ 425— λ 445 having been made, the same series of coincidences was found as in the case of the Norwegian rutile (see the fifth and sixth columns of the table), and hence at the same time the fact was demonstrated that Swedish rutile also contains vanadium.

* "Ueber das Vorkommen des Vanads in den Skandinavischen Rutilitarten," *Bihang till Svenska vetensk. Akad. Handl.*, xxii., Afd. 1, No. 7. From the *Astrophysical Journal*, v., No. 3.

Vanadium.		Rutile I.		Rutile II.		Remarks.
λ	i.	λ	i.	λ	i.	
4033'03	1'2	..	..	..	..	Also a weak line in Ti.
90'70	3	90'73	1	..	..	Coincident, belong to Va. All the rutile lines are weaker on the comparison plate than here represented.
92'87	3	92'83	1'2	..	..	
95'60	3	95'65	1	..	..	
4100'00	3'4	4099'94	1'2	..	..	
05'30	3	05'31	1'2	..	..	
09'95	3'4	09'92	1'2	..	..	
12'00	4	11'91	2'3	..	..	
15'30	3'4	15'32	2	..	..	
16'65	3	16'64	1'2	..	..	
23'65	3	23'68	2'3	..	..	
28'25	3'4	28'20	2	..	..	Divided. λ Ti $>$ λ Va.
31'35	1	31'38	1'2	..	..	Coinc. Belongs to Va.
34'60	3'4	34'60	1'2	..	..	Divided. λ Ti $<$ λ Va.
59'87	2'3	59'79	2'3	..	..	Coinc. Belongs to Va.
69'45	1'2	69'46	2	..	..	Divided. λ Ti $<$ λ Va.
83'45	..	83'45	1'2	..	..	Clearly divided. λ Ti $>$ λ Va.
4227'95	2	27'80	2	..	..	Probably divided. λ Ti $>$ λ Va.
68'85	3	..	1	..	1	Widely separated.
71'80	3	..	1	..	1	
4330'15	3	..	1—	..	1+	
33'00	3	..	1—	..	1+	
41'15	3	..	1+	..	1'2	
53'05	3'4	53'01	1	53'01	1'2	All these lines occur on the rutile photographs with the observed intensities. The blanks in the columns 3 and 5 of wave-lengths indicate that these lines do not occur in my catalogue of the titanium lines. On the plates here investigated they occur with the given intensities.
79'45	4'5	79'40	2	79'40	3	
84'90	4'5	84'85	2	84'85	2'3	
90'15	4'5	90'11	2	90'15	2+	
95'40	4	..	2	..	2	
4400'75	4	00'74	1'2	00'74	2	
06'85	4'5	..	1'2	..	2	
07'90	4'5	07'85	1'2	07'85	2+	
08'40	4	08'39	1'2	08'39	2+	
08'65	4'5	08'70	1'2	08'70	2+	
16'65	3	16'70	2	16'70	2	Clearly divided. λ Ti $>$ λ Va. Perhaps. λ Ti $>$ λ Va. Probably divided. λ Ti $>$ λ Va.
41'90	3'4	41'86	1'2	41'86	..	
44'40	3'4	44'41	3	44'41	..	

If, further, the observed intensities of the vanadium lines which occur in the photographic spectra of the two kinds of rutile are compared, it will be seen that the intensities for Rutile II., obtained at K ringsbr cka, are greater throughout the spectrum than those for Rutile I. Since this fact appears to indicate that Swedish rutile contains a greater amount of vanadium than Norwegian, it was of interest to test the relation of the two varieties in this respect by a special experiment, in which the exposure and development were exactly the same. With this object, two exposures in the upper region of the spectrum were made on the same plate, using for each a different half of the slit, with electrodes which in one case were made of Norwegian and in the other case of Swedish rutile. The exposure was in each case 1'5 minutes. The developed plate showed the titanium lines with identically the same intensity in both spectra, while the vanadium lines were considerably stronger in the spectrum of the Swedish rutile. In order that this fact may be clearly brought out, I have made a photographic copy of a drawing, in which the appearance of the negative under the microscope of the measuring engine is represented with all possible exactness. By this it will be seen that vanadium lines of the Norwegian rutile have a distinctly lower intensity, so that, in fact, some of the weakest of them fail to appear.

From what has been given above, I believe that it may be regarded as proved that both kinds of rutile contain vanadium, the Norwegian as well as the Swedish, but that the Swedish variety contains a considerably greater amount of this metal than the other. Whether this amount of vanadium is great enough to be recognised or quantitatively determined by ordinary chemical analysis is a question for the solution of which the above experiments afford no data, or at least only such as are highly

uncertain, for we have as yet no trustworthy information as to the sensitiveness of the spectral reactions of the elements.

On the plates which contain the spectra of the two kinds of rutile, the correspondence of lines (leaving out of consideration the difference of intensity of the vanadium lines already mentioned) is complete, with one exception. This exception is found in three quite strong lines which occur in the spectrum of the Swedish rutile, but of which there is scarcely a trace in the other variety. By referring their positions to neighbouring titanium lines I obtained for these lines the following wave-lengths:—

$$\lambda = 4254'50 \\ 74'90 \\ 89'90$$

while the strongest lines in the whole chromium spectrum have, according to my earlier measures, the wave-lengths

$$\lambda = 4254'49 \\ 74'91 \\ 89'87$$

The lines therefore belong to chromium, the presence of which is thereby demonstrated, and this result is in agreement with the analysis of Ekeberg.

Probable Antiquity of Mining for Tin in Bretagne. —L. Davy.—All authors who have studied the ancient working of tin in the west of Europe admit that it was far anterior to the occupation of the country by the Romans, and think that the mines of Abbaretz-Nozay were abandoned by the Gauls about the date of the Roman invasion.—*Comptes Rendus*, cxxv., No. 5.

A CRITICAL REVIEW OF THE METHODS
OF DETERMINING MINERALS.*By Dr. JOSEPH W. RICHARDS,
Of the Department of Metallurgy and Mineralogy of the Lehigh
University.

THE determining of a mineral consists in finding out its species, and in doing this so conclusively as to prove that it is the species named to the exclusion of all other similar related minerals.

The question must be met at the outset, "What constitutes a mineral species?" I will answer by quoting our one-time professor of chemistry at the Institute, Dr. Persifer Frazer, whose word in geology and mineralogy carries with it the weight of an authority. Says Dr. Frazer, "Every true mineral is a definite chemical compound or element, homogeneous throughout its parts, and capable of expression in a formula. Its molecule is a distinctive whole—the unit of its mass—and incapable of division as long as the mineral retains its characteristic properties."

It is one of the easiest things in the world to accept the present scientific definition, and to forget the laborious steps by which such an apparently simple conclusion was evolved. Dr. Frazer's definition appears to us so nearly self-evident that it is difficult to realise that eighty years ago mineralogists were hotly debating the question as to whether the identity of a mineral consisted in its chemical composition or in the sum total of its physical properties. Thence arose two schools of mineral classification. Werner and his followers adopted the natural-history method of classification on the basis of physical properties alone, and prided themselves on having achieved a classification entirely independent of any aid from chemistry. The other school, of which Berzelius was one of the founders, insisted on the chemical composition as the basis of a proper classification, but were not so exclusive as the other school, and admitted physical distinctions, particularly that of form (*i.e.*, crystallisation), into their smaller subdivisions.

It will help, in our further consideration of the subject, to examine more closely these two schemes, particularly the Wernerian.

Cronstedt, about 1750, divided the mineral kingdom into four great divisions, viz.:—

- | | |
|----------------------|--------------------|
| I. Earth and stones. | III. Combustibles. |
| II. Salts. | IV. Ores. |

Werner, the first of the illustrious teachers of mineralogy at Freiberg, adopted this division and extended it greatly. He divided the first order into nine genera, as follows:—

- | | |
|----------------|----------------|
| <i>Earths.</i> | |
| 1. Zirconian. | 5. Calcareous. |
| 2. Siliceous. | 6. Barytic. |
| 3. Aluminous. | 7. Strontian. |
| 4. Magnesian. | |
| <i>Stones.</i> | |
| 8. Diamond. | 9. Hallites. |

Here, indeed, it would appear that the classification was nothing less than purely chemical, yet it was not. A certain number of external characters which siliceous minerals, for instance, usually exhibit being assumed as generic characters, or as a type of the genus, every mineral possessing those characters, whether it contains any silic or not, is arranged under the siliceous genus. And so with the other of the nine genera. Under these principles, Werner saw no inconsistency in placing sapphire in the siliceous genus and opal in the aluminous. I need not expatiate on the absurdity of this procedure from our modern standpoint, but it is only the stupendous development of chemical investigation in recent years that has lifted mineralogy out of that confusion.

Salts, according to Werner, included only such minerals as have some taste and a considerable degree of solubility in water; and they were divided into carbonates, nitrates, muriates, and sulphates.

Combustibles were divided into sulphur, bitumen, graphite, and resin.

Ores were divided into as many genera as there were distinct metals found as ores. Thus, all the iron ores constituted one genus. The genera were divided into species, depending on their external characters. This was a true chemical basis to start with, but far inferior to the classification according to the acid ingredient instead of the base.

The key-note of this system is found in the following quotation from Werner himself:—"Whenever the external characters and the chemical composition are *at variance*, the species is determined solely by the *external characters*." According to this method of reasoning, gypsum and selenite were classed as different species because they looked different, although of exactly the same composition, while calamine and smithsonite were classed together as one mineral because they looked alike, although of very different composition.

Coming to the chemical systems of classification, they were far from being above reproach, yet they showed fewer inconsistencies than the other method. Many mistakes were made, it is true, principally owing to the imperfect state of chemical science. The phenomena of dimorphism were yet awaiting the light of Mitscherlich's genius for their explanation, and the reproach was cast on the chemical method that it had to classify together calcite and aragonite as one species, while everybody knew they were different. Many were the hours spent by zealous adherents of the chemical side, in trying to establish by analysis some essential difference between calcite and aragonite, and thus to silence their opponents. Mitscherlich's researches on isomorphism and dimorphism, however, cleared up so many of these difficult points that in 1841 Rammelsberg was able to resuscitate the almost defeated chemical method, and to champion it so effectively as to have made it the present accepted basis of mineral classification.

A very striking example of this change of base in mineral classification is shown in the successive standard American works on mineralogy. Shepard's work, issued in 1835, 1844, and 1857, in successive editions, was on the natural-history method. J. D. Dana's first edition, in 1837, followed the same system; the second edition, in 1844, was similar, but described in an appendix Rammelsberg's chemical system; the third edition, in 1850, discarded the natural-history method of classification, and adopted altogether the chemical method. This has been retained in all subsequent editions of this magnificent work, which has become not only the American authority on mineralogy, but, one may almost say, the international authority. The importance of this classification to our discussion merits that it be given at once, in outline, as follows:—

Dana's Classification of Minerals.

- I. Native elements.
- II. Sulphides, arsenides, antimonides, selenides, and tellurides.
- III. Chlorides, bromides, and iodides.
- IV. Fluorides.
- V. Oxygen compounds.
 - (1) Oxides.
 - (2) Ternary oxygen compounds, or oxygen salts.
 1. Silicates.
 2. Tantalates and columbates, &c.
 3. Phosphates, arsenates, vanadates, &c.
 4. Borates.
 5. Tungstates, molybdates, chromates, &c.
 6. Sulphates.
 7. Carbonates.
- VI. Hydrocarbons.

* The Journal of the Franklin Institute, cxliv., p. 139.

It is now proper to inquire into the subject of the identification of a mineral, i. e., its determination.

The question is how to most quickly, easily, and surely identify an unknown specimen. This is not exactly the same sort of question as the one of classifying properly a mineral after it is completely described; they are two aspects of the same problem, but from different points of view; for, as Dr. Sterry Hunt has said, "A natural system of classification is not subordinate to the end of identifying species, but should consider objects in all their alliances and relations." Systems for identifying species are not therefore necessarily along the identical lines of mineral classification. However, the natural-history method of classification by outward characters adapts itself very readily to schemes of identification in which the physical properties are given the pre-eminence in determining the mineral. Determinations of hardness, streak, lustre, gravity, fusibility, form, &c., are easily made without any special training, and therefore are most useful to beginners in the science of determining minerals. The chemical system of classification, however, at once suggests chemical analysis as the true basis of a determinative method, and lends itself most kindly to that means of identification. Moreover, the methods of chemical analysis, particularly those of blowpipe analysis, have developed coincidentally with the growth of the chemical classification of minerals.

The postulate may be made, that if every mineral species is a definite chemical compound, then the true basis of identifying the mineral should be the chemical analysis. In many cases this may not be the quickest way, but it is bound to be the surest. But shall we throw away all the aid to be derived from physical tests? By no means. If from the properties at once evident on casual inspection memory suggests a familiar mineral, let chemical analysis at once come in to confirm or deny the determination. *In the great majority of cases the physical determination is practically valueless without the analytical confirmation; and there are very few cases in which the chemical confirmation is absolutely unnecessary or useless.* On the other hand, whenever a mineral is not at once recognised on inspection, the true basis of search to determine it should be to fix as nearly as possible its composition by chemical analysis, and then to utilise physical tests to distinguish between minerals difficult or impossible to separate by differences in their composition.

Tables based entirely on physical tests have been constructed; mixed tables, based on a combination of both physical and chemical tests, have been devised; I have preferred to attack unknown minerals *entirely* from the chemical side, and will later on explain what appear to me the advantages of this method.

It is, of course, superfluous to analyse in detail every one of the numerous schemes of determinative mineralogy which have been constructed; it will suffice to select a few typical examples, and to discuss them on general principles.

Dana gives in an appendix to his text-book on Mineralogy one set of tables based primarily on the crystal system, then on lustre (metallic and non-metallic), and then with the minerals arranged according to specific gravity. This classification is very useful if the mineral shows clearly its crystallisation, and is truly of the system which it appears to be; but every mineralogist knows that minerals are oftener uncrystallised than crystallised, and that in probably one case out of three the system is not what it appears to be. The question of lustre may sometimes mislead. The specific gravity may be depended upon in probably three cases out of four. Summing up the probabilities that a mineral could be identified by the use of such a table, they are about one in two if the specimen is crystallised, and not more than one in ten if it is uncrystallised. In scarcely any case would the identification be completely satisfactory without the chemical confirmation.

A set of tables in more extensive use than Dana's, and

based also on physical tests alone, is that of Dr. Weisbach, well known in English by Dr. Frazer's translation. The following outline shows the scheme of this classification:—

Weisbach's Tables.

- I. Minerals with metallic lustre.
 1. Red. 2. Yellow. 3. White. 4. Grey. 5. Black.
- II. Minerals of sub-metallic or ordinary lustre, and with coloured streaks, as follows:—
 1. Black. 2. Brown. 3. Red. 4. Yellow. 5. Green. 6. Blue.
- III. Minerals of ordinary lustre, with white or grey streak.
 1. Very soft. 2. Soft. 3. Semi-hard. 4. Hard. 5. Very hard.

Supplementary table for Class III.:—

- A. Soluble in water.
- B. Effervescing in HCl (carbonates).
- C. Not soluble in water or containing CO₂.
 1. Containing water—Easily fusible to infusible.
 2. Anhydrous—Easily fusible to infusible.

In using these tables, if there is no question as to the lustre, or colour, or streak or hardness, that is, if all these properties are normal and observed correctly, the specimen thereupon falls into a class varying in number from five to sixty-three, among which it is to be distinguished by a critical comparison of its other physical properties. In the case of the class of five, this is an easy matter; in the majority of cases, the classes averaging thirty to fifty, this is an arduous or impossible task. We have further to consider that sometimes the lustre is doubtfully metallic, frequently the colour is not easy to judge, that the streak is often misleading, and, most of all, the hardness very seldom to be relied upon. I should say in all fairness, taking all these points into consideration, that a careful observer would not place more than fifty out of one hundred promiscuous specimens in the class to which Weisbach assigns them, and out of those fifty correctly classed, would not be able to identify over twenty-five with any reasonable degree of certainty without having recourse to the chemical composition for confirmation.

One of the best examples of tables based on mixed physical and chemical tests is that of Von Kobell, which is the basis of the classification so well known and largely used in the United States, viz., Brush's. They are as follows:—

Von Kobell's Tables.

- I. Metallic lustre.
 - a. Native malleable metals and mercury.
 - A. Fusible at 1 to 5 or easily volatile, containing—
 1. Arsenic. 2. Selenium. 3. Tellurium. 4. Antimony. 5. Sulphur. 6. None of these.
 - B. Fusible above 5, or infusible, not volatile.
 1. Containing manganese. 2. Magnetic B.B. 3. All others.
 - II. Without metallic lustre.
 - A. Easily volatile or combustible.
 - B. Fusible at 1 to 5.
 - (I.) Reduces to metallic button, or magnetic B.B.
 1. Silver. 2. Lead. 3. Copper {with As. no As.
 4. Cobalt. 5. Magnetic B.B. 6. All others.
 - (II.) Not belonging to (I.)
 1. Alkaline after ignition.
 2. Soluble in HCl, without residue.
 3. Soluble in HCl, with gelatinous residue.
 4. Soluble in HCl, with sandy residue.
 5. Insoluble in HCl, has Mn.
 6. All others.
 - I. Metallic lustre.
 - II. Without metallic lustre.
 - A. Easily volatile or combustible.
 - B. Fusible at 1 to 5.
 - C. Fusible above 5, or infusible.

1. With cobalt nitrate show alumina.
2. With cobalt nitrate show zinc.
3. React alkaline after ignition.
4. Soluble in HCl, contain no silica.
5. Soluble in HCl, contain silica.
 - (1). Hydrous.
 - (2). Anhydrous.
6. Not belonging to the foregoing divisions.
 - (1). Hardness under 7.
 - (2). Hardness 7 or over.

It will be seen that the basis of this classification is primarily and secondarily physical, afterwards chemical composition plays an important part. The superiority of this method over the previous ones based solely on physical tests, *lies almost altogether in the extent to which the chemical tests are introduced.* There is still some uncertainty as to lustre being correctly determined, and considerable uncertainty is introduced by the factor of fusibility where it lies anywhere near to five; but, granting that these are correctly determined, then the chemical tests are reached and the uncertainties mostly cease. No one worthy of being called a chemist can mistake sulphur for selenium or silver for lead; the identification of such characteristic elements is practically a certainty. With such chemical tests as solubility in hydrochloric acid, with a gelatinous or sandy residue or no residue at all, there is still considerable room for uncertainty, and the scheme is weak in proportion as they are introduced.

Dr. Fuchs introduced mixed tables based primarily on chemical composition, and to that extent superior to Weisbach's. As a whole I have found these tables (or subsequent ones based upon them) to be the most satisfactory tables published. To observers skilled in blowpipe analysis they furnish the most satisfactory means of identification of an unknown specimen. The scheme is as follows:—

Fuchs's Tables.

(By the Blowpipe).

- I. Heated B.B. on charcoal.
 1. Volatilises or burns.
 2. Arsenic fumes { metallic.
non-metallic.
 3. Selenium fumes. 4. Shows antimony.
 5. Shows tellurium.
 6. Reacts alkaline { soluble.
non-soluble.
 7. Residue magnetic.
- II. Reduced B.B. with soda, on charcoal.
 1. Reacts for sulphur and gives metallic button.
 2. Reacts for sulphur and gives no metallic button.
 3. No sulphur reaction, but gives metallic button.
- III. Shows manganese in the beads { metallic lustre.
non-metallic lustre.
- IV. Shows zinc with cobalt solution.
- V. Soluble in HCl, without residue.
 1. Fusible B.B. (a). Hydrous. (b). Anhydrous.
 2. Infusible B.B. (a). Hydrous. (b). Anhydrous.
- VI. Soluble in HCl to a jelly (silica).
(Classified similarly to V.)
- VII. Soluble in HCl, with separation of silica (no jelly).
 1. Hydrous. 2. Anhydrous.
- VIII. Insoluble in HCl, but bead test shows silica.
 1. Fusible B.B. 2. Infusible B.B.
- IX. Belonging to none of the previous divisions.

The weakest point of these tables is in the action of hydrochloric acid, where there is room for some uncertainty, and where tests for the elements, as in the previous divisions, are generally much more satisfactory. Division IV. should also be abolished, and placed as a fourth subdivision of II., for by reducing with soda and a little borax on charcoal, zinc is more certainly identified than by the cobalt nitrate test. Cadmium minerals could also have been included under the same subdivision. Taking these tables altogether, I would call them the most satisfactory of the published tables.

(To be continued).

NOTICES OF BOOKS.

Incompatibilities in Prescriptions; for Students in Pharmacy and Medicine, and Practising Pharmacists and Physicians. By EDSSEL A. RUDDIMAN, Ph.M., M.D. New York: John Wiley and Sons. London: Chapman and Hall, Lim. 1897. Pp. vi.—267. 8vo.

THE author of this valuable work was the Professorship of Pharmacy and Materia Medica in Vanderbilt University, Nashville, Tennessee, and, having the Degrees of Master in Pharmacy and Doctor of Medicine, is well qualified for his undertaking. There are two distinct divisions of the book. Part I. contains, in a condensed form, the more common incompatibilities, the substances being arranged in alphabetical order of their Latin names. Part II. contains 325 prescriptions, with a criticism of each explaining the difficulties of compounding, or the objections to the form in which they are written. In the first part the ordinary reactions of the given substance are stated, and the decompositions liable to ensue when brought into contact with other substances by trituration, and aqueous or alcoholic solution.

The several reactions in each paragraph are numbered for convenience of reference; thus *antipyrinum* is treated in twenty-three short items, and *acidum tannicum* in twenty-six items.

Carelessness on the part of the medical man, or on the part of the apothecary's assistant, may cause explosions when certain substances are triturated in a mortar or brought together in solution; the active substances may be entirely changed by oxidation, reduction, or double decomposition; an insufficiency of a solvent may interfere with the desired form of prescription; the order in which ingredients are mixed may greatly affect the result; the proportion of the substances may have to be altered to obtain the end sought; and some ignorant practitioners may actually send to the pharmacist prescriptions which cannot be filled without danger to the compounder or to the patient. All these cases are abundantly illustrated and carefully explained in the second part of the book, by citing actual prescriptions and criticising them. Prescription No. 36 calls for potassium chlorate, precipitated sulphur, sulphide of antimony, and sugar, and the author remarks that each substance should be powdered separately and mixed lightly, which is certainly a wise caution. Prescription No. 132 contains amyl nitrite, potassium iodide, alcohol, and syrup of lemons, with the direction *Cito dispensetur!* Dr. Ruddiman recommends that this should not be dispensed as written, but that the physician should be communicated with. Prescription No. 87 is said to be part of the Keely cure for alcoholism; it contains chloride of gold and sodium, sulphate of strychnine, sulphate of atropine, and fluid extract of cinchona.

The author suggests that the student of pharmacy study each prescription thoroughly before consulting the note accompanying it.

The work shows care throughout, but the author overlooks the formation of sodium nitrate in his note to prescription No. 170. Two indices complete the well-printed volume, one to incompatibilities and one to prescriptions.

It would be interesting to learn the sources of the prescriptions cited by the author; they are presumably from the note-books of apothecaries and from published medical treatises, a list of which he gives as the authorities consulted. To the lay mind many of them appear to be horrible mixtures, almost as objectionable as the nostrums of Paracelsus and his followers; and the question arises, Does the physician who prescribes substances that mutually decompose when mixed (sometimes generating bodies quite different from their original forms) take into consideration the physiological action of the newly-formed compounds, or does he attribute the effects to the primary ingredients?

This volume will be invaluable to students of pharmacy and to young practitioners; but is not its existence a commentary on the deficient education in the fundamental truths of chemistry of those licensed to compound and to administer medicines to a long-suffering people?

H. C. B.

Sessions of the Superior Board of Health, corresponding to the Year 1896. ("Sesiones del Consejo Superior de Higiene Publica, correspondientes al Año de 1896"). Santiago de Chile.

CONSIDERABLE attention seems to have been paid to the potable waters of Valparaiso. There have been in that city many cases of typhoid fever and of acute intestinal catarrh; the public water-supply passes through seats of infection. It was pronounced necessary that Santiago should be seivered; that it is not possible to use for the disinfection of the sewage-waters the chemical procedures already in use, and that it is impossible to disinfect all the irrigation-waters of Santiago.

A Detailed Course of Qualitative Chemical Analysis of Inorganic Substances, with Explanatory Notes. By ARTHUR A. NOYES, Ph.D., Assistant Professor of Chemistry at the Massachusetts Institute of Technology. Third Edition. London and New York: Macmillan and Co., Lim. 1897. 8vo., pp. 89.

WE have here what may be regarded as an abridgment of the well-known treatise on Qualitative Analysis by the late Prof. Remigius Fresenius. We can find little, if anything, to which we have any right to object. It seems to us slightly strange to find the author recommending that the laboratory work should be accompanied by *recitations* in which the process of analysis is discussed in detail.

CORRESPONDENCE.

MODERN ALCHEMY.

To the Editor of the Chemical News.

SIR,—As you have allowed Dr. Bolton to indulge in a little modern scientific witch-finding at my expense, I hope you will permit me to make the following rejoinder:—

1. My accuser says that I have "with boldness, publicity, and persistency," made claim to "success in transmutation or creation of gold." This charge is not in accordance with the facts of the case. Dr. Bolton is sufficiently well versed in modern newspaper methods to know that publicity frequently takes place in opposition to the desires of the parties concerned. He knows that this has largely been so with regard to my work respecting the interchangeability of the so-called "elements." He knows that the average reporter makes a surprising hash of scientific statements, and that the average catchpenny newspaper editor takes reprehensible liberties even with signed communications and interviews. He knows that when I was applied to respecting the accuracy or otherwise of the report printed in the *New York Journal*, I declined to admit that it was more than "substantially correct"—a fact which surely should have precluded such report being quoted in your columns as my "own words."

2. The following correspondence has passed between my accuser and myself, viz.:—

a. Bolton to Emmens, June 21st, 1897:—

"I am preparing for an English paper an account of your work of converting silver into *argenteum*, and write to enquire whether you can send me any later publication of your own than your announcement made in August, 1896.

"I will be obliged for any circular or other publication you have issued in respect to Ar."

b. Emmens to Bolton, June 22nd, 1897:—

"Replying to your letter of yesterday, I have to say that it will give me much pleasure to send you additional information. The little pamphlet will be ready in a few days, and then I will mail you a copy. It will, I think, interest you, even if you be not a Unitarian in material philosophy."

c. Bolton to Emmens, July 11th, 1897:—

"Your polite note was duly received, and I shall be very glad to receive a copy of your forthcoming pamphlet as soon as issued."

d. Emmens to Bolton, July 13th, 1897:—

"Yours of the 11th has reached me, and I now have the pleasure of sending you by this mail a copy of the just-completed pamphlet, '*Arcana Naturæ*'; and inasmuch as reference is made therein to my '*Argentaurum Papers No. 1*,' I also enclose a copy of that book."

e. Bolton to Emmens, July 16th, 1897:—

"I acknowledge with thanks your letter of July 13th, and your publications received to-day. I had heard of your work on Gravitation, but had not seen it, and shall examine it with interest.

"The '*Arcana Naturæ*' gives me just the information I needed. Thanking you for your courtesy, I am, &c."

The pamphlet alluded to in these letters is not a publication for sale. I prepared it in order to save time and trouble in replying to the numerous enquiries that are addressed to me from all parts of the world. It contains an explanation as to various developments and modifications that have taken place in my gold-work since the original announcement in the New York newspapers. Yet, with this explanation staring him in the face, my accuser, under the guise of reporting "*Recent Progress of Alchemy in America*," has served up, for the information of your eminent circle of readers, a mere copy of a popular article in a sensational newspaper published on August 18th, 1896, and has suppressed the particulars with which I civilly furnished him at his own request.

3. The pamphlet contains a clear and explicit account of a process of producing from Mexican dollars a substance that will pass muster as gold. My accuser withholds this from your readers, and then goes on to charge me with "secret processes" and "vagueness of description."

4. The pamphlet contains more than one explicit confession of my ignorance of natural things. Yet my accuser charges me with "an assumption of esoteric knowledge expressed in pseudo-philosophic language."

5. My accuser charges me with "an imperative present need of gold." In one sense of the words it may be conceded that every scientific man, as well as every scientific institution, is in need of gold. But this is not the sense in which Dr. Bolton wrote. No reader can be so obtuse as not to understand the "fling"; it is a piece of personal abuse for which my accuser cannot adduce one jot or tittle of justification. I have not appealed to him or to the public for one cent. I have, on the contrary, made him and other correspondents a free gift of information which has cost me many thousands of dollars to acquire. I think your readers will be of opinion that the learned gentleman owes it to them and himself to tender me an apology.

6. I will conclude with the narration of what may perhaps be deemed an interesting historical fact. On March 16th, 1897, I sent to the United States Assay Office, in New York, a Mexican dollar to be tested for gold. The report was *nil*. Thinking that this might have been an accidental case of individual purity, I then sent four Mexican dollars to the same Assay Office and had them there cut into halves. One set of halves was officially assayed with the same *nil* result as before. The other set was treated

in the Argentaurum Laboratory, without the addition of gold in any form, and the result was a relatively considerable production of a metal which answered to all the usual tests of gold, and was subsequently purchased as gold by the U.S. Assay Office.

For my own mental satisfaction I then instituted enquiries at the Assay Office as to the precise meaning to be attached to a report of *nil*; and I was informed that, where small weights of bullion are concerned, the report means that the amount of gold is less than one part in ten thousand—a proportion which, of course, was vastly exceeded by the gold actually produced by my treatment of the four half-dollars. One of two alternative conclusions was therefore compulsory:—Either some of the silver or copper in the dollars had been changed into gold or its simulacrum by my treatment; or the gold already existed in the dollars and was separated by my treatment, though not by the treatment in vogue at the U.S. Assay Office. I make every chemist a present of the dilemma. For my own part my "assumption of esoteric knowledge" does not carry me so far as to make me believe myself a more skilful assayer of bullion than Mr. Andrew Mason and his assistants.—I am, &c.,

STEPHEN H. EMMENS.

Argentaurum Laboratory,
20, Central Avenue, New Brighton,
Staten Island, New York, U.S.A.,
August 16, 1897.

[A specimen of argentaurum sent me by Dr. Emmens has been examined in the spectrograph. It consists of gold with a fair proportion of silver and a little copper. No lines belonging to any other known element, and no unknown lines, were detected.—W. C.]

HYPONITROUS ACID.

To the Editor of the Chemical News.

SIR,—As you have published in the CHEMICAL NEWS a translation of Hautzsch and Kaufmann's paper upon Hyponitrous Acid, which appeared in *Liebig's Annalen*, you will, I am sure, permit me to point out, also in the CHEMICAL NEWS, some errors which have entered into that paper, through its authors' neglect to read the literature of the subject.

Hautzsch and Kaufmann say that Maumené discovered hyponitrous acid. The facts are, however, as I have fully shown in the *Annalen*, that I am the undisputed discoverer, and that Maumené never experimented on the subject at all, and actually denied the existence of the acid, on purely theoretical grounds (CHEM. NEWS, xxiii., 206; xxv., 153 and 285).

The method for preparing hyponitrous acid, which the authors call Zorn's, and claim to have perfected (with what propriety will be shown some other time), is my method, my original method,—that is, for I have, in conjunction with Haga, published two others.

Piloty's method, referred to by the authors, had been, seven years before, anticipated by Haga and me (*Journ. Chem. Soc.*, lv., 760) in a much simpler and less expensive form, with the same satisfactory yield of more than half the theoretical quantity. In place of benzo-sulphydroxamic acid, we had used sulphydroxamic acid (oxamidodisulphonic acid); that was all the difference. The equation in our case was—

$2\text{NaO} \cdot \text{SO}_2 \cdot \text{NH} \cdot \text{OH} + 4\text{KOH} = 2\text{NaO} \cdot \text{SO}_2 \cdot \text{OK} + (\text{KON})_2$,
and that in Piloty's case,—

$2\text{C}_6\text{H}_5 \cdot \text{SO}_2 \cdot \text{NH} \cdot \text{OH} + 4\text{KOH} = 2\text{C}_6\text{H}_5 \cdot \text{SO}_2 \cdot \text{OK} + (\text{KON})_2$.

Concerning ammonium hyponitrite the authors should properly have referred to me as authority rather than to Zorn, since I had stated so many years earlier (CHEM.

NEWS, xxiii., 208) that ammonium chloride and silver hyponitrite give silver chloride and an alkaline solution which at once evolves ammonia.

The induction of the reaction of a hyponitrite with ferrous sulphate as a nitrate or nitrite by contact with undiluted sulphuric acid, which is made a point of by the authors, had already been recorded by me.

There are two important erroneous experimental observations contained in the paper, which the authors would have saved themselves from making had they become sufficiently acquainted with the literature of their subject before publishing, but the consideration of these I reserve till I publish the sequel to my first paper.

I wish to state that the correctness of the contents of this letter is not disputed. Professor Hautzsch and also Mr. Piloty have each in turn courteously acknowledged their oversight, and Professor Hautzsch has, without my action, recognised one of the errors as to experimental fact above alluded to.

It is the re-appearance of Hautzsch and Kaufmann's paper in the CHEMICAL NEWS which has obliged me to publish this letter. Also, to prevent misconception, I will add that no work upon hyponitrous acid—and there has been much—surpasses, if it equals, in value that done by the excellent Russian chemist Zorn, who died very young, to the great loss of chemistry.—I am, &c.,

EDWARD DIVERS.

Imperial University of Tokyo,
July 4, 1897.

PS. The number of the CHEMICAL NEWS which contains the beginning of Hautzsch and Kaufmann's paper has also a letter from Mr. Thomas Christie in commendation of taka-diastase, so successfully discovered and studied by Mr. Takaminé, F.C.S. I take this opportunity to express the pleasure I feel in the fact that Mr. Takaminé is the first Japanese pupil I ever had, and that he pursued chemistry under me for four years.—E. D.

THE ALLEGED NEW ELEMENT IN IRON.

To the Editor of the Chemical News.

SIR,—I was much interested in Mr. Boucher's article in the last issue of the CHEMICAL NEWS (vol. lxxvi., p. 99), describing a possible element in pig-iron, and as I have come across a constituent of steel during the last few months resembling in every respect the description of its properties as given by Mr. Boucher, it may be of some interest if I give my experience.

A few months ago I received a small sample of steel drillings which had originally come from the Continent, and in the course of an analysis, in which, beside the ordinary constituents, appreciable quantities of arsenic, copper, cobalt, and nickel were found, I separated a dark brown sulphide in the arsenic group which had all the properties as described by Mr. Boucher—the most remarkable of them being the beautiful blue colour on evaporating down with sulphuric or hydrochloric acid. I had a very limited amount of the steel, and was working on 5 grms. After repeated precipitation and purification of the sulphide to eliminate any arsenic and antimony, the precipitate weighed 0.017 grm.; showing a very considerable proportion of this element—a quantity much too great to neglect. I therefore wrote to my clients requesting them to send me a further sample, as the steel contained a rare element which I wished to investigate further; this they were unable to do, and so the matter has stood in abeyance. The experiments I performed resembled closely those detailed by Mr. Boucher, with the exception that I used nitro-hydrochloric acid to dissolve the steel, instead of sulphuric acid.

At first sight it appeared to resemble molybdenum, but this was contradicted by many of its reactions, and I shall

await Sir William Crookes's report with much interest.—
I am, &c.,

FREDK. G. RUDDOCK.

Laboratory and Assay Office,
19, Stanley Street, Warrington,
August 31, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 5, August 2, 1897.

Outset of the Combination between Hydrogen and Oxygen.—M. Berthelot.

Analysis of Aluminium and its Alloys.—Henri Moissan.

Fixation and Nitrification of Nitrogen in Arable Soils.—P. P. Dehérain.

Poisoning by the Sweat of a Healthy Man.—S. Arloing.—This paper shows that sweat contains substances energetically poisonous, possessing analogies with the certain microbial toxins.

On the Symmetric Tetramethyldiamidodiphenyl-dianthranaltetramethyldiamide of the corresponding Oxanthranol.—A. Haller and A. Guyot.—The conditions in which we have operated, the composition of the substance which we have obtained, enable us to conclude that we are in the presence of the diamidodimethylamido-phenyloxanthranaldimethyl.

Atomic Weights of Nitrogen, Chlorine, and Silver.—A. Leduc.—The author admits the following atomic weights:—O=16 (base), N=14.005, H=1.0076, Cl=35.470, Ag=107.916. As for sulphur we deduce from the experiments of Sias the atomic weight 32.056, he adopts this number, although the experiments of Dumas lead to 31.86, and those of Erdmann and Marchand to 32.005.

Thermo-chemical Determinations relating to the Cupric Compounds.—Paul Sabatier.—A thermo-chemical paper not suited for useful abstraction.

On certain Bromo-ketones.—A. Collet.—The author describes the parabromopropionyltoluene, the parabromobutyryltoluene, the bromopropionylparaxylene, the bromopropionylphenyl, and the chloride of dibromophenylpropionyl.

Observations on the Conjunction of the Diazo-derivatives with the Phenols.—Ch. Gassman and Henry George.—Not suitable for useful abstraction.

Carbinose.—J. Effront.—Carbinose appears as a syrupy substance, not crystallisable, soluble in water and alcohol, and answering to the formula $C_6H_{12}O_6$. It ferments readily with beer yeasts. The rotatory power of carbinose, its melting-point, and the crystalline form of its combinations with phenylhydrazin distinguish these sugars from the other monosaccharides.

On an Organic Compound rich in Manganese extracted from the Woody Tissue.—G. Guérin.—This compound, as obtained from beech-wood, yielded per cent —C, 52.762; H, 5.04; N, 4.60; S, 0.666; P, 1.297; Mn, 0.402.

Moniteur Scientifique,
Series 4, Vol. xi., Part 1.

Progress realised in our Knowledge of the Constitution of the Alkaloids of Quinine.—Ch. Gassmann.—Not suitable for abstraction.

On the Applications of Electrolysis to Organic Chemistry.—L. Gourwitsch.—Already inserted.

Part 2.

On the rôle played by Peroxides in the Phenomena of Slow Oxidation.—A. Bach.—Already inserted.

Buletinul Societatii de Stiinta din Bucuresti
(Bulletin of the Scientific Society of Bucharest),
No. 2, 1897.

The Secretary-General, in a short obituary notice, announces the lamented death, after a painful illness, of C. Gogu, Professor of Mathematics in the University of Bucharest, and President of this Society, and he bears tribute to the loss the Society has sustained in his death. A biographical sketch of his life work will be printed in the *Annales of the Society*.

Subterranean Water in the North-west region of Bucuresilor.—N. Cucu St.—The author has sunk a number of shafts in various parts of the district, and has found water in considerable quantity in several places. He fully describes all the works carried out and gives excellent illustrations of some of the "fountains."

On a very Sensitive Reaction for Nitric Acid.—E. Riegler.—Reprinted from the *Pharmaceutischen Centralhalle*, Nos. 13 and 14, 1897.

Notes on the Constitution and Classification of the Sulpho-arsenical, Sulpho-antimonial, and Sulphobismuthic Minerals.—V. C. Butureanu.—There is, as is well known, an important group of minerals in nature known generally as sulpharsenides, sulphantimonides, and sulphbismuthides. The author has examined all of these that he has been able to obtain, and classified them according to their chemical formula, expressed graphically.

Classification of the Crystalline Rocks of the Central Zone of the Roumanian Carpathians.—L. Mrázec.—The rocks of this district are divided into three groups. The first comprises the distinctly crystalline rocks of the granitic gneiss type; the second, those of the well-crystallised mica-schists; and the third, the slightly crystalline chloritic schists. The second group is the one the author deals principally with in this paper.

ERRATUM.—P. 75, col. 1, line 32 from top, for "0.00366" read "0.00365."

UNIVERSITY COLLEGE, BRISTOL, CHEMICAL DEPARTMENT.

Professor—SYDNEY YOUNG, D.Sc., F.R.S.
Lecturer—FRANCIS E. FRANCIS, B.Sc., Ph.D.
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TO MANUFACTURING CHEMISTS.

THE LONDON COUNTY COUNCIL is prepared to receive Tenders for the supply and Delivery at the Barking and Crossness Outfall Works of 2500 or 5250 Tons of PROTO-SULPHATE OF IRON (Commercial Green Vitriol).

Persons desiring to submit Tenders may obtain the form of tender, and other particulars, on application at the Engineer's Department, County Hall, Spring Gardens, S.W. Tenders must be upon the official forms, and the printed instructions contained therein must be strictly complied with. Separate prices are to be quoted for the 2500 tons and the 5250 tons of Proto-Sulphate of Iron. Tenders are to be delivered at the County Hall in a sealed cover addressed to the Clerk of the London County Council, and marked "Tender for Proto-Sulphate of Iron. No tender will be received after 10 a.m. on Tuesday, the 5th day of October, 1897. Any tender which does not comply with the printed Instructions for Tender may be rejected.

The Council does not bind itself to accept the lowest or any tender, and it will not accept the tender of any person or Firm who shall on any previous occasion have withdrawn a tender after the same has been opened, unless the reasons for the withdrawal were satisfactory to the Council.

Spring Gardens, S.W.,
September 1, 1897.

C. J. STEWART,
Clerk of the Council.

TO LIME MERCHANTS AND OTHERS.

THE LONDON COUNTY COUNCIL is prepared to receive Tenders for the supply and delivery at the Barking and Crossness Outfall Works of 11,500 or 23,000 Tons of LIME.

Persons desiring to submit Tenders may obtain the form of tender, and other particulars, on application at the Engineer's Department, County Hall, Spring Gardens, S.W. Tenders must be upon the official forms, and the printed instructions contained therein must be strictly complied with. Separate prices are to be quoted for the 11,500 tons and 23,000 tons of Lime. Tenders are to be delivered at the County Hall in a sealed cover addressed to the Clerk of the London County Council and marked "Tender for Lime." No tender will be received after 10 a.m. on Tuesday, the 5th day of October, 1897. Any tender which does not comply with the printed Instructions for Tender may be rejected.

The Council does not bind itself to accept the lowest or any tender, and it will not accept the tender of any person or Firm who shall on any previous occasion have withdrawn a tender after the same has been opened, unless the reasons for the withdrawal were satisfactory to the Council.

Spring Gardens, S.W.,
September 1, 1897.

C. J. STEWART,
Clerk of the Council.

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AND

JOURNAL OF PHYSICAL SCIENCE.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1972.

(STUDENTS' NUMBER).

A WORD TO STUDENTS.

IN spite of the recognised, and we might almost say the boasted, superiority of German chemists in the quantity of research yearly executed, and in their number of investigators, our Continental rivals have not the slightest disposition to rest and be thankful. In a pamphlet by Prof. Dr. Ferd. Fischer we find recommendations for further extending and securing their position in technical chemistry as compared with that of other countries. The author has here collected the views of his colleagues on the subject in question, and lays them before the world.

We find that there are in Germany four thousand technical chemists, exclusive of about two hundred others who study chemistry as a pure science. We cannot ascertain whether the figure of four thousand includes German technical chemists living and practising in other countries. We suspect that this is not the case. At most of the technical schools it is proposed to extend the curriculum, and to introduce examinations as a test for the proficiency of the students. Whether, and in how far, these proposals will lead to results of practical value time alone can decide. It will be noted that the course of study in the polytechnics and technical high schools varies greatly.

In many instances subjects are introduced which, however important in themselves, seem to us unnecessary in the special training of a technical chemist. We must keep in mind that, even for the most able and the most industrious student, there are only twenty-four hours in the day, and further that the great principle of division of labour must never be overlooked.

Instruction in law, with the single special exception of patent law, seems to us a needless encroachment on the spheres of the solicitor and the barrister.

We perceive that in some departments the candidate has a choice of subjects. Thus at Berlin, in the examination in chief, the student may select (a) geology, (b) spectrum analysis or general metallurgy.

We think that we in Britain are recovering some of the lost ground; but an immense field lies before us. Above all things we have to fight against the claims of the word-mongers; they are still far too strong and too influential to admit of our taking the foremost position in the world of Science.

UNIVERSITIES AND COLLEGES.

UNIVERSITY OF LONDON.

CANDIDATES for any Degree in this University must have passed the Matriculation Examination. No exemption from this rule is allowed on account of Degrees obtained or Examinations passed at any other University. This and all other Examinations of the University, together with the Prizes, Exhibitions, Scholarships, and Medals depending upon them, are open to Women upon exactly the same conditions as to Men.

There are two Examinations for Matriculation in each year; one commencing on the second Monday in January, and the other on the second Monday in June.

The Examination is conducted by means of Printed Papers; but the Examiners are not precluded from putting, for the purpose of ascertaining the competence of the candidates to pass, *viva voce* questions to any candidate in the subjects in which they are appointed to examine. These Examinations may be held not only at the University of London, but also, under special arrangement, in other parts of the United Kingdom, or in the Colonies.

Every candidate for the Matriculation Examination must, not less than five weeks before the commencement of the Examination, apply to the Registrar for a Form of Entry, which must be returned not less than four weeks before the commencement of the Examination, accompanied by a Certificate showing that the candidate has completed his sixteenth year, and by his Fee for the Examination. As no candidate can be admitted after the List is closed, any candidate who may not have received a Form of Entry within a week after applying for it must communicate immediately with the Registrar, stating the exact date of his application and the place where it was posted.

Every candidate entering for the Matriculation Examination for the first time must pay a Fee of £2 to the Registrar. If a candidate withdraws his name, or fails to present himself at the Examination, or fails to pass it, the Fee shall not be returned to him, but he shall be allowed to enter for any subsequent Matriculation Examination upon payment, at every such entry, of an additional Fee of £1, provided that he comply with the Regulations in the preceding paragraph.

Candidates are not approved by the Examiners unless they have shown a competent knowledge in each of the following subjects:—Latin. Any one of the following Languages:—Greek, French, German, Sanskrit, or Arabic. The English Language, and English History, with the Geography relating thereto. Mathematics. Mechanics. One of the following branches of Science:—Chemistry, Heat and Light, Magnetism and Electricity, Botany.

The Examination in Chemistry is—Chemistry of the Non-metallic Elements; including their compounds, their chief physical and chemical characters, their preparation, and their characteristic tests.

A Pass Certificate, signed by the Registrar, will be delivered to each successful candidate after the Report of the Examiners has been approved by the Senate.

If in the opinion of the Examiners any candidates in the Honours Division of not more than twenty years of age at the commencement of the Examination possess sufficient merit, the first six among such candidates will receive an Exhibition of thirty pounds per annum for the next two years; the second among such candidates will receive an Exhibition of twenty pounds per annum for the next two years; and the third will receive an Exhibition of fifteen pounds per annum for the next two years; such exhibitions are payable in quarterly instalments provided that on receiving each instalment the Exhibitioner declares his intention of presenting himself either at the two Examinations for B.A., or at the two Examinations for B.S., or at the Intermediate Examination in Laws, or at the Preliminary Scientific M.B. Examination, and Intermediate Examination in Medicine, within three academical years from the time of his passing the Matriculation Examination.

Under the same circumstances, the fourth among such Candidates will receive a prize to the value of ten pounds in books, philosophical instruments, or money; and the fifth and sixth will each receive a prize to the value of five pounds in books, philosophical instruments, or money.

Any candidate who may obtain a place in the Honours Division at the Matriculation Examination in January is admissible to the Intermediate Examination either in Arts or in Science in the following July.

INTERMEDIATE EXAMINATION IN SCIENCE.

The Intermediate Examination in Science will commence on the third Monday in July.

No candidate (with the exception of such as have obtained Honours at the Matriculation Examination in the preceding January) is admitted to this Examination within one academical year of the time of his passing the Matriculation Examination.

The Fee for this Examination is £5.

Examination for Honours.

Candidates for Honours in Chemistry will be examined in Inorganic Chemistry, treated more fully than in the Pass Examination. In addition, they will be examined practically in Simple Qualitative Analysis. This Examination will consist of six hours' examination by two printed papers and of six hours' practical work.

In the Examination for Honours, the Candidate, not being more than 22 years of age at the commencement of the Pass Examination, who most distinguishes himself will receive an Exhibition of £40 per annum for the next two years.

B.Sc. EXAMINATION.

The B.Sc. Examination will be held on the third Monday in October.

Candidates for this Examination are required to have passed the Intermediate Examination in Science at least one academical year previously.

The Fee for this Examination is £5.

Examination for Honours.

The examination for Honours in Chemistry will take place on Monday, Tuesday, and Wednesday in the week following the Examination for Honours in Mathematics; on Monday by printed papers (chiefly on Organic Chemistry), and on Tuesday and Wednesday by practical examination in Qualitative and Quantitative Analysis.

The candidate, being not more than 23 years of age, who most distinguishes himself in Chemistry, will receive £50 per annum for the next two years, with the style of University Scholar.

DOCTOR OF SCIENCE.

The examination for the Degree of Doctor of Science takes place annually within the first twenty-one days of June.

No candidate is admitted to the examination for the Degree of D.Sc. until after the expiration of two Academical Years from the time of his obtaining the Degree of B.Sc. in this University.

Every candidate for this Degree must state in writing the special subject within the purview of the Faculty of Science, as set out in the Programme of the B.Sc. Examination, upon a knowledge of which he rests his qualification for the Doctorate; and with this statement he shall transmit an original Dissertation or Thesis (at least six copies), printed, type-written, or published in his own name, treating scientifically some special portion of the subject so stated, embodying the result of independent research, or showing evidence of his own work, whether conducted independently or under advice, and whether based on the discovery of new facts observed by himself, or of new relations of facts observed by others, or, generally, tending to the advancement of Science. Every candidate may further specify any printed contribution or contributions to the advancement of Science which he has at any time previously published. If the Dissertation or Thesis be approved by the Examiners, the candidate shall be required to present himself at the University upon such day or days within the first twenty-one days of June as may be notified to him, and shall, at the discretion of the Examiners, be further tested, either orally or practically, or by printed questions or by all of these methods, with reference both to the special subject selected by him and to the Thesis.

PRELIMINARY SCIENTIFIC (M.B.) EXAMINATION.

This Examination takes place twice in each year,—once, for Pass and Honours, commencing on the third

Monday in July; and once for Pass Candidates only, commencing on the third Monday in January.

No candidate shall be admitted to this Examination unless he shall have passed the Matriculation Examination. Not less than five weeks before the commencement of the Examination he must apply to the Registrar for a Form of Entry, which must be returned not less than four weeks before the Examination, accompanied with the candidate's fee.

The Fee for this examination is Five Pounds.

UNIVERSITY OF OXFORD.

Waynflete Professor of Chemistry—W. Odling, M.A., F.R.S.

Every Student must reside in one or other of the Colleges or Halls, or in licensed lodgings, for a period of three years. Students of Chemistry can obtain the degree of B.A. by passing preliminary examinations in Arts and in Science, and a final Honour examination in Chemistry. Chemistry may also be taken as part of the examination for a Pass degree. Graduates of other Universities suitably qualified can obtain the degree of Bachelor of Science after an approved course of study or research and two years' residence.

University Laboratory.—Demonstrators, W. W. Fisher, V. H. Veley, F.R.S., J. E. Marsh.—The fee for students working in the Laboratory for three days in the week during the Term is £3; for students working every day, £5.

Christ Church Laboratory.—A. Vernon Harcourt, F.R.S. Scholarships of about the value of £80 are obtainable at the majority of the colleges, by competitive examination in Natural Science.

More detailed information may be obtained from the Examination Statutes; the Student's Handbook to the University; and from the professors and college tutors.

UNIVERSITY OF CAMBRIDGE.

Professor of Chemistry—G. D. Liveing, M.A., F.R.S.

Jacksonian Professor of Natural and Experimental Philosophy—J. Dewar, M.A., F.R.S.

The Student must enter at one of the Colleges or Hostels, or as a Non-collegiate Student, and keep terms for three years by residence in the University. He must pass the previous examination in Classics and Mathematics, which may be done in the first or third term of residence, or, through the Oxford and Cambridge Schools Examination Board, or through the Senior Local Examinations, before commencing residence. He may then proceed to take a Degree in Arts, either continuing mathematical and classical study, and passing the ordinary examinations for B.A., or going out in one of the Honour Triposes.

The scholarships, ranging in value from £20 to £100 a year, are chiefly given for mathematical and classical proficiency. Scholarships, or Exhibitions, are given for Natural Science in King's, Trinity, St. John's, St. Peter's, Clare, Trinity Hall, Queen's, Jesus, Christ's, Sidney, Pembroke, Caius, and Downing Colleges; the dates of the examinations vary, but are always fully advertised.

The Chemical Laboratory of the University is open daily for the use of the Students. The Demonstrators attend daily to give instructions. A list of the lectures is published annually, in June, in a special number of the *Cambridge University Reporter*, which may be had from the Cambridge Warehouse, in Paternoster Row, or through any bookseller.

Non-collegiate Students are allowed to attend certain of the College Lectures and all the Professors' Lectures, and have the same University status and privileges as the other Students. Full particulars may be obtained by forwarding a stamped directed envelope to the Assistant Registrar, Cambridge, or from the *Cambridge University Calendar*.

UNIVERSITY OF DUBLIN.
TRINITY COLLEGE.

Professor of Chemistry—J. Emerson Reynolds, D.Sc., M.D., F.R.S.

Assistant Lecturer—Emil A. Werner, F.C.S., F.I.C.

Demonstrator—J. Percy Bailey, B.A.

The general Laboratories include working accommodation for 120 Students, and the Quantitative and Research Laboratories for about 40 Students. The Laboratories will open on the 1st of October. Lectures will commence about November 1st.

The Laboratories and the Lectures of the Professor of Chemistry can now be attended by Students who do not desire to reside in the University or proceed to its Degrees.

The full Course of General and Analytical Chemistry occupies three years, but a Student is free in his third year to devote most of his time to a special department of Pure or Technical Chemistry. Students can enter for any portion of the Course. The following Lectures are delivered:—

1. *Inorganic Chemistry and Chemical Philosophy*.—Elementary, first year; advanced, second year.
2. *Organic Chemistry*.—General, second year; advanced, third year.
3. *Metallurgy*.—A Course for Engineering and Technical Students.

The Laboratories are open every day from 10 to 5 o'clock (except Saturdays, when they close at 1 o'clock).

The Summer Course of Practical Chemistry for Medical Students begins during the first week in April and terminates with the first week in July.

The University of Dublin grants the Degree of Doctor of Science to graduates of Master's standing whose independent researches in any branch of Science are of sufficient merit.

KING'S COLLEGE.

(DIVISION OF ENGINEERING AND APPLIED SCIENCE).

Professor of Chemistry—J. M. Thomson, F.R.S., F.C.S.

Demonstrator of Practical Chemistry—Herbert Jackson, F.C.S.

Assistant Demonstrators—P. H. Kirkaldy, F.C.S., and W. H. Sodeau, B.Sc.

The Academical Year consists of Three terms. The days fixed for the Admission of New Students in the Academical Year 1897-98 are September 30, January 13, and April 27.

Students of the First Year are admitted to the Course of Theoretical and Applied Chemistry. The Course commences with a view of the conditions suitable for the production of Chemical Phenomena, after which the laws of Chemical Attraction are discussed, and the Non-metallic Elements and their principal compounds are described. The Metals and their principal compounds are next examined, care being taken to point out the applications of the Science to the Arts; and the processes of the different Manufactures and of Domestic Economy are explained and illustrated. Examinations of the Class, both *viva voce* and by written papers, are held at intervals during the course at the usual Lecture hour.

Second Year.—Students attend in the Laboratory twice a week, and they go through a course of Manipulation in the most important operations of Chemistry, including the first steps of Analysis. Any Student of this Division may be admitted to this Class at any period of his study on payment of an extra fee.

Experimental and Analytical Chemistry in the Laboratory.—The object of this Class is to afford to Students who are desirous of acquiring a knowledge of analysis, or of prosecuting original research, an opportunity of doing so under the superintendence of the Professor and Demonstrator; Students may enter, upon payment of extra fees, at any time except during the vacation, and for a period of one, three, six, or nine months, as may best suit their convenience. The laboratory hours are from ten till

four daily, except Saturday, on which day the hours are from ten till one.

In addition to the Laboratory Fee, each Student defrays the expenses of his own experiments. The amount of this expense, which is comparatively trifling, is entirely under his own control.

Special hours and fees are arranged for the convenience of such Third Year Students as wish to study Analytical Chemistry.

Fees.—Chemistry per term, £3 3s. od.; per ann., £8 8s. od.; Practical Chemistry per term, £4 4s. od.; per ann., £10 10s. od.; Experimental and Analytical Chemistry—Daily attendance: One month, £4 4s.; Three months, £10 10s.; Six months, £18 18s.; Nine months, £26 5s. Three days a week: One month, £2 12s. 6d.; Three mos., £6 6s.; Six mos., £11 11s.; Nine mos., £15 15s.

METALLURGY.

Professor—A. K. Huntington, F.I.C., F.C.S., &c.

The following subjects are treated of in the Lectures: The Selection and Economic Preparation of Fuel and of Refractory Materials; the methods by which metals are obtained from their ores, and the means by which they are rendered suitable for the various requirements of the Arts.

Particular attention is paid to the study of the Nature and Properties of Metals and Alloys available for Constructive Purposes.

In the Metallurgical Laboratory, which is always open during College hours, the relation between the Chemical Composition of Metals and their Mechanical Properties may be studied by the aid of Testing Machinery.

PHOTOGRAPHY.

Lecturer—Prof. J. M. Thomson, F.R.S., F.C.S.

In addition to the regular College Course in Photography occasional classes may be formed. For further particulars application should be made to Prof. Thomson.

EVENING CLASSES.

Classes for Evening Instruction in various subjects are held during the months from October to March, inclusive, and during the months of April, May, and June.

UNIVERSITY COLLEGE.

FACULTY OF SCIENCE.

Professor—William Ramsay, Ph.D., F.R.S.

Assistants—Morris Travers, B.Sc., Alexander Kellas, B.Sc., and J. W. Walker, M.A., Ph.D.

The Session is divided into three Terms, as follows, all the dates being inclusive:—

First Term, from Tuesday, October 5th, until Friday, December 17th;

Second Term, from Tuesday, January 11th, 1898, till Friday, April 1st;

Third Term, from Tuesday, April 26th, till Tuesday, July 5th. Class Examinations begin on June 22nd.

Junior Courses of Inorganic Chemistry.

First Term: Tuesday, Thursday, and Saturday at 10. Second and Third Terms: Tuesday, Thursday, and Saturday. Fee:—£4 4s.

These Courses will each consist of about thirty lessons, partly theoretical and partly practical, on the non-metallic elements. Frequent exercises will be given.

Senior Course of Inorganic Chemistry.

First and Second Terms: The Class meets four times a week, on Mondays, Wednesdays, Fridays, and Saturdays, at 9, for Lectures. Examinations, and Exercises.

Fees:—For the Course, £7 7s.; Perpetual, £9 9s.; for the First or Second Terms, £4 4s.

This Course and the Practical Class cover the subject as prescribed for the Preliminary Scientific (M.B.) and Int. Examination in Science of the University of London.

For the Preliminary Scientific Examination Students who take the three subjects for that examination in July attend during the First and Second Terms.

Advanced Course of Chemistry.

Second and Third Terms.—The class meets twice a

week, on Tuesdays and Thursdays, at 9, beginning on January 13. The hour will be altered by special arrangement with the class if necessary.

Fee:—For the Course, £3 3s.; for a Term, £2 2s.

This Course will be found suitable for those about to proceed to graduation as Bachelor of Science in London University, and to those who intend to choose Chemistry as a profession. Such students should also work in the Laboratory during as many hours as they can spare.

Organic Chemistry.

Tuesday, Thursday, and Saturday, at 9, in the First Term; Tuesday, Thursday, and Saturday, at 10, in the Second Term; and Tuesday and Thursday at 9, and Saturday at 11, in the Third Term. The hour of meeting will be altered should the class desire it.

This Course of Organic Chemistry is intended for those who are studying the subject from a scientific standpoint. Candidates for Honours at the Int.M.B. are, however, recommended to attend this Course besides the Special Summer Course.

The Course includes the subjects required at the B.Sc. Examination, Pass and Honours; but no previous acquaintance with Organic Chemistry will be expected of those joining the Class.

Fee:—For the Course, £6 6s.; for the Second and Third Terms, £4 14s. 6d.; for a Term, £2 12s. 6d.; for a Second Course, £3 3s.

Practical Classes.

Practical Classes in Inorganic and Organic Chemistry are conducted by the Assistants.

Analytical and Practical Chemistry.

The Laboratory is open daily from 9 a.m. to 4 p.m., Saturdays excepted, from October until the middle of July, with a short recess at Christmas and at Easter.

Fees: for the Session, £26 5s.; six months, £18 18s.; three months, £10 10s.; one month, £4 4s.

Three specified days a week:—for the Session, £15 15s.; six months, £11 11s.; three months, £6 6s.; one month, £2 12s. 6d., exclusive of expense of materials. Students may enter at any period of the Session.

The Laboratory Course includes the Practical Chemistry required at the following Examinations of the University of London:—Prel. Sci. (M.B.), Intermediate M.B., Intermediate Science, B.Sc.

Students who wish to attend the Lectures on Chemical Technology may acquire here the requisite knowledge of Practical Chemistry and Analysis.

When accompanied by, or preceded by, attendance on the Lectures on Inorganic and Organic Chemistry, the Laboratory Course qualifies Students in the application of Chemistry to Manufactures, Metallurgy, Medicine, or Agriculture, &c.

There is also a Chemical Library containing the chief Journals and Standard Works on Chemistry.

Certificates of Honour are granted to competent Students on the work done during the Session. The Tuffnell Scholarship (£100 for two years) will also be competed for in the Session 1897-98; also the Cloth-worker's Scholarship of £30.

ROYAL COLLEGE OF SCIENCE AND ROYAL SCHOOL OF MINES.

Professor—W. A. Tilden, D.Sc., F.R.S.

Assistant Professor—W. P. Wynne, D.Sc., F.R.S.

Demonstrators—H. Chapman Jones and J. W. Rodger, A.R.C.S.

Assistants—G. S. Newth, A. Eiloart, Ph.D., B.Sc., and M. O. Forster, Ph.D.

The Royal College of Science at South Kensington is intended, primarily, for the instruction of teachers, and of students of the industrial classes selected by competition in the examinations of the Science and Art Department. The Royal School of Mines is incorporated with the Royal College of Science. Students entering for the Associateship of the Royal School of Mines obtain their general scientific

training in the Royal College of Science. The instruction in the Royal College of Science is arranged in such a manner as to give the Students a thorough training in the general principles of Science, followed by advanced instruction in one or more special branches of Science. The Associateship is granted in certain divisions or lines of study. Students who go through any one of the prescribed courses of instruction and pass the necessary Examinations receive a Certificate of Associateship of the Royal College of Science, or of the Royal School of Mines. Students who are not candidates for the Associateship are permitted to enter as occasional students in one or more special branches of science, and on passing the examination receive a Certificate to that effect. The Associateship of the Royal College of Science is given in one or more of the following divisions:—Mechanics, Physics, Chemistry, Biology, Geology, and Agriculture, and the Associateship of the Royal School of Mines in Metallurgy and Mining.

The course of instruction, which lasts for three years, is the same for all the divisions during the first year, after which it is specialised in accordance with the Scheme detailed in the Prospectus of the School.

The Session is divided into two Terms. The first Term begins on the 7th of October and ends about the middle of February. The second Term begins in the middle of February and ends about the middle of June.

Examinations are held at the end of each course of instruction and at such other periods as may be found necessary. On the results of these examinations the successful candidates are arranged in two classes, first and second. There are also "Honours" examinations for the subjects of the third year, the successful candidates being placed in order of merit. A student obtains the Associateship who passes in all the subjects of the first two years and in the third year those of the special division he selects for his Associateship. A student who goes through the prescribed course of instruction in any subject and passes the final examination in it receives a certificate to that effect.

Students who do not wish to attend the lectures are admitted for short periods to the laboratories, at the discretion of the Professors. The fees for the laboratories are £4 per month.

Students not entering for the Associateship are admitted to any particular course of study, so far as there is room, on payment of the fees shown in the following table:—

	Lectures.	Laboratory.
Chemistry	3	13
Physics	5	12
Biology with Botany ..	5	12
Geology with Mineralogy ..	4	8
Mechanics	4	6
Metallurgy	2	13
Mining	4	
Astronomical Physics ..	2	3

Agricultural Chemistry, per term, £13. Mathematics and Mechanical Drawing, £3 per term. Model and Free-hand Drawing, £1 per term. Descriptive Geometry, £3 per session. Mine Surveying, £10.

The fees for the first two years amount to about £75, and for the remainder of the course for the Associateship they vary from £30 to about £40.

Both the private and the State-aided students are required to furnish themselves with certain instruments and apparatus before the commencement of the courses. These are enumerated in the syllabuses of the several subjects.

Officers of the Army, Navy, and Civil Service, recommended by their respective Departments, are admitted to the Lectures and Laboratories at half fees.

Associates of the Royal College of Science or of the Royal School of Mines have the privilege of free admission to the Library and to all the courses of lectures.

Bona fide teachers qualified to earn payments for teaching Science according to the rule of the Science and

Art Directory may obtain permission to attend free any course of lectures.

Several valuable Exhibitions, Scholarships, and Prizes are attached to the studentship.

Summer Courses for Teachers.—Short courses of instruction are given annually, about July, in different branches of science for the benefit of teachers of science schools in the country. The courses last three weeks. About 250 teachers are admitted to them, and they receive third class railway fare to and from South Kensington, and a bonus towards their incidental expenses of £3 each. (See Science and Art Directory.)

Working Men's Lectures.—Notification of these will be given in the newspapers.

THE SCHOOL OF THE PHARMACEUTICAL SOCIETY OF GREAT BRITAIN.

The Fifty-sixth Session will commence on Monday, October 4th, 1897.

Professors—Chemistry, J. Norman Collie, Ph.D., F.R.S.; Botany, J. Reynolds Green, Sc.D., F.R.S., F.L.S.; Materia Medica and Pharmacy, Henry G. Greenish, F.I.C., F.L.S. (Dean).

A Course of Lectures on Physical, Inorganic, and Elementary Organic Chemistry commences in October and terminates at the end of June. An Advanced Course of Lectures begins in October and extends to the end of March. These Lectures are adapted to the requirements of Pharmaceutical and Medical Students, and also those who are proceeding to degrees at the University of London, or who are preparing for the examinations of the Institute of Chemistry.

Entries may be made for single classes. Certificates of attendance at the two Courses of Lectures on Chemistry and at the Chemical Laboratories are accepted as evidence of chemical training by the Institute of Chemistry in connection with the Examinations for the Associateship, and also by the conjoint Board of the Royal Colleges of Physicians and Surgeons, as well as by other examining bodies.

Prospectuses and further information may be obtained from Mr. Richard Bremridge, Secretary and Registrar, 17, Bloomsbury Square, London, W.C.

UNIVERSITY COLLEGE OF WALES, ABERYSTWYTH.

UNIVERSITY OF WALES.

Professor—H. Ll. Snape, D.Sc. (Lond.), Ph.D. (Göttingen), F.I.C.

Assistant Lecturer and Demonstrator—A. W. Warrington, M.Sc. (Vic.), F.I.C.

Lecturer in Agricultural Chemistry—J. Alan Murray, B.Sc. (Edin.).

The College is open to male and female students above the age of sixteen years. The Session commences on Tuesday, October 5, on which day all Students will be expected to meet the Professors in the Examination Hall of the College.

Lecture Courses.—(1) Matriculation Course; three lectures weekly during the Michaelmas and two weekly during the Lent and Easter Terms. (2) Intermediate Science Pass Course; four lectures weekly during the Lent and Easter Terms. (3 and 4) B.Sc. Courses; A, three lectures weekly on Organic Chemistry; B, two lectures weekly on Chemical Theory. (Courses A and B will generally be given in alternate Sessions; for 1897-8, Course A.) (5 and 6) Courses in Agricultural Chemistry. For students in their first year, 3 lectures, and for those in their 2nd year, 2 lectures weekly throughout the Session.

Laboratory Courses.—The Laboratory is open daily from 10 a.m. to 1 p.m., and from 2.15 to 5 p.m., except on Wednesdays and Saturdays. Classes for the Systematic Study of Qualitative and Quantitative Analysis will be formed, and Special Courses will be

arranged for those who intend to follow Medicine or Pharmacy, or any one particular branch of Applied Chemistry, always provided that such Students possess the requisite knowledge of Theoretical Chemistry. The hours will be arranged, as far as possible, to suit the requirements of the individual Student.

The College is recognised by the Royal University of Ireland, and by the Colleges of Physicians and Surgeons of England, Scotland, and Ireland as an institution at which the instruction necessary for their respective Diplomas in Medicine, in Chemistry, Physics, and Biology may be given. One year for graduation in Medicine and two years for graduation in Science may be spent at Aberystwyth.

Fees.—The Fee for the whole Session, if paid in advance, is £10; if paid by Single Terms, for the first term of attendance in each Session, £4; for the second term, £3 10s.; for the third term, £3. These composition fees enable the Student to attend any or all the Classes of the College, with the exception that a small extra fee is charged for Laboratory Instruction. Thus, for Practical Chemistry, the additional fee is, for six hours' work per week, 10s. per term, and for twelve hours, 20s. per term. The fees for those who desire to spend several days weekly in the laboratory may be learned on application to the Registrar. Fee for a single Lecture Course £1 per term.

Scholarships and Exhibitions varying in value from £10 to £40 per annum will be offered for competition at examinations which commence on September 21, and exhibitions are awarded at the end of the Session on the results of the class examinations.

The Chemical Laboratories in connection with this College have been recently built, and are fitted with every convenience for the prosecution of chemical studies.

Intending Students requiring further information are recommended to write to the Registrar for a copy either of the General Prospectus or of one of the Special Prospectuses issued for the Agricultural and Normal Departments.

UNIVERSITY COLLEGE OF NORTH WALES, BANGOR.

A CONSTITUENT COLLEGE OF THE UNIVERSITY OF WALES.

Chemistry.—Professor, James J. Dobbie, M.A., D.Sc. Demonstrator, Fred. Marsden, Ph.D., B.Sc. Assistant Lecturer in Agricultural Chemistry, F. V. Dutton.

Physics.—Professor, Andrew Gray, M.A., LL.D., F.R.S.

The Session opens October 5th, 1897. All regular classes are open to men and women students above the age of 16 years. The following Courses of Lectures will be given.

Matriculation Course.—Subjects: Those prescribed for the Matriculation Examination of the University of Wales. Fee for the Term £2 2s. A class for revision of Matriculation Work will be held during the Summer Term. Fee for the Term, £1 1s.

Intermediate Course.—Inorganic Chemistry and Elementary Physical Chemistry. Fee for the Term £2 2s.

B.Sc. Course.—Advanced Inorganic Chemistry. Fee for the Session, £3 3s.

Medical Course.—Fee, £4 4s.

Agricultural Chemistry.—Fee, £2 2s.

Laboratory Courses.—The laboratory is open on five days of the week from 10 a.m. to 4 p.m. for instruction in Chemical Analysis and in the Application of Chemistry to Medicine and the Industrial Arts. Fees: six hours per week, £1 1s. per Term; twelve hours, £2 2s.; eighteen hours, £3 3s.; twenty-four hours, £4 4s. Composition Fee for all Laboratory Classes of the Intermediate Science Course taken in one year, £4 4s.

The Chemistry, Botany, Zoology, and Physics Courses are recognised for Medical graduation in the Universities of Edinburgh and Glasgow, and students can make one *Annus medicus* at the college. The Science Courses are

recognised for part of the science degree course of the University of Edinburgh.

UNIVERSITY COLLEGE OF SOUTH WALES AND MONMOUTHSHIRE, CARDIFF.

Professor—C. M. Thompson, M.A., D.Sc., F.C.S.

Demonstrators—E. P. Perman, D.Sc., F.C.S., and A. A. Read, F.I.C., F.C.S.

The Session commences October 4th, and terminates on June 24th, and is divided into three terms.

The Junior Course (delivered during the Michaelmas term only) consists of about 50 lectures, and will cover the subjects prescribed for the Matriculation examinations of the University of Wales and the University of London. Fee, £2 2s. A revision class is held in the Summer term.

The Intermediate Course consists of about 80 lectures held during the Lent and Summer terms in continuation of the Junior Course, and is the qualifying course for the Intermediate Examination of the University of Wales. Together with laboratory practice, it will cover the subjects required for the Intermediate Examination in Science and the Prel. Sci. (M.B.) Examination of the University of London. Fee, £4 4s.

The Senior Course consists of some 90 lectures on Organic Chemistry; Fee, £3 3s.

A course of 20 lectures on Qualitative Analysis and a short course on Organic Chemistry will also be given.

The following lectures on Metallurgy will be given by Mr. Read:—10 lectures on Fuel; Fee, 10s. 6d. 20 lectures on General Metallurgy; Fee, £1 1s. 30 lectures on the Manufacture of Iron and Steel; Fee, £1 1s. A practical course on Iron and Steel Analysis will also be held, and practical instruction in Dry Assaying will be given in the Metallurgical Laboratory, which is fitted with the necessary furnaces and apparatus.

In the laboratory each student works independently, so that the course of study may be adapted to the requirements of the individual. Hours, 9 to 1 and 2 to 5; Saturday, 9 to 1. Fees—Six hours per week, £3 3s. per session; twelve hours, £2 2s. per term; eighteen hours, £3 3s. per term; twenty-four hours £4 4s. per term.

Registered medical students can prepare for the Intermediate M.B. Examination of the University of London, and spend three out of their five years of medical study in Cardiff. Medical students wishing to graduate at a Scottish University, or preparing for a Conjoint Board Surgical and Medical Diploma, or for the Diploma of the Society of Apothecaries, can spend two years in Cardiff. For further information see the prospectus of the Faculty of Medicine, which may be obtained from the Registrar.

The College is recognised as an institution at which two years of the course for the degree of Bachelor of Science of the University of Edinburgh may be spent.

Students by making a payment of £10 at the commencement of each session may compound for all lecture fees for the whole session. Laboratory fees are not included in the composition fee, but Students preparing for the Science Examinations of the University of Wales and of the University of London may, by making a payment of £13 13s. at the commencement of each Session, compound for both Lecture and Laboratory Fees during the Session.

At the entrance examination in September, and the annual examination in June, several scholarships and exhibitions are awarded. Great importance is attached to special excellence in one subject.

The College Prospectus, and also further information as to scholarships, may be obtained from the Registrar.

A Hall of Residence for Women Students is attached to the College.

UNIVERSITY COLLEGE, BRISTOL.

Professor of Chemistry—Sydney Young, D.Sc., F.R.S.

Lecturer—Francis E. Francis, B.Sc., Ph.D.

The session 1897-98 will begin on October 5th. Lectures and classes are held every day and evening throughout

the Session. In the Chemical Department lectures and classes are given in all branches of theoretical chemistry, and instruction in practical chemistry is given daily in the chemical laboratory. The department of experimental physics includes various courses of lectures arranged progressively, and practical instruction is given in the physical and electrical laboratories. The Department of Engineering and the Constructive Professions is designed to afford a thorough scientific education to students intending to become engineers, or to enter any of the allied professions, and to supplement the ordinary professional training by systematic technical teaching. This department includes courses specially arranged for students intending to become civil, mechanical, electrical, or mining engineers, surveyors, or architects. Those who attend the mechanical engineering course enter engineering works during the six summer months, and, in accordance with this scheme, various manufacturing engineers in the neighbourhood have consented to receive students of the College into their offices and workshops as articled pupils at reduced terms. Medical education is provided by the Faculty of Medicine of the College. Several Scholarships are tenable at the College. Full information may be obtained from the Secretary.

DAY LECTURES.

Inorganic Chemistry.

The Courses treat of the principles of Chemistry, and of the Chemistry of the Non-Metals and Metals.

Junior Course.—Two Lectures a week will be given during the First and Second Terms. Fee, £3 3s.

Special Course.—A special course of Lectures is also given to Engineering Students.

Senior Course.—Three Lectures a week will be given throughout the Session. Fee, £5 5s. There will be tutorial classes in connection with the Junior and Senior Courses.

Advanced Course.—One Lecture a week will be given throughout the Session. Fee, £2 12s. 6d.

Organic Chemistry.

This Course will relate to the more important groups of the Compounds of Carbon.

Two Lectures a week will be given during the Second Term, and three Lectures a week during the Third Term. Fee, £3 3s. An advanced course of lectures will also be given one day a week during the session. Fee, £2 12s. 6d.

Practical Chemistry.—Laboratory Instruction.

The Laboratory will be open daily from 10 a.m. to 5 p.m., except on Saturdays, when it will be closed. Instruction will be given in the Laboratory in all branches of Practical Chemistry, including Qualitative and Quantitative Inorganic and Organic Analysis, the preparation of Chemical Products, and Inorganic and Organic Research. Special facilities will be afforded to those who desire to study Practical Chemistry as applied to the different processes employed in the Arts and Manufactures. Fees in Guineas—

	5 Days a Week.	4 Days a Week.	3 Days a Week.	2 Days a Week.	1 Day a Week.
Per Session ..	15	12½	10	7½	5
„ Two Terms ..	11	9	7½	5½	3½
„ One Term ..	7	6	4½	3½	2½

Students may arrange to divide their days of laboratory work into half-days.

Chemical Scholarship.—Among others, a Chemical Scholarship of £25 is offered for competition.

EVENING LECTURES.

Two courses of Lectures will be delivered during the First and Second Terms; they will be devoted to the consideration of the general Principles of Chemistry and Chemical Physics and the Chemistry of Non-Metallic and Metallic Elements. Special attention will be paid throughout to those products which have a practical application in the Arts and Manufactures. Fee for each course, 7s. 6d.

Practical Chemistry.—Laboratory Instruction.—The

Laboratory will be open on Tuesday and Wednesday evenings from 7 till 9. Instruction will be given in Qualitative and Quantitative Analysis, and in the Preparation of Chemical Products. Fees:—(Two Terms) Two Evenings, 25s.; One Evening, 15s. (One Term) Two Evenings, 15s.; One Evening, 10s 6d.

University College, Bristol, has been approved by the Council of the Institute of Chemistry as a College at which all the subjects required for the admission of Associates to the Institute are taught.

The Calendar of the College, price 1s. (post-free, 1s. 4d.), containing detailed information of the various Courses, may be obtained on application to the Secretary.

MASON COLLEGE, BIRMINGHAM.

Professor—Percy F. Frankland, Ph.D., B.Sc., F.R.S.

Assistant Lecturer—C. F. Baker, Ph.D., B.Sc.

Demonstrator—W. R. Innes, Ph.D., M.Sc.

The Session will be opened on September 30th, 1897.

Elementary Course.

Forty Lectures adapted to the requirements of beginners will be given in the Winter and Spring Terms. Lecture days—Wednesdays and Fridays at 11.30.

Persons entirely unacquainted with Chemistry are recommended to attend this Course before entering for the General Course. Candidates for the Matriculation Examination of the University of London also are advised to attend this Course.

General Course.

The General Course of Lectures on Chemistry will be found useful by Students who are afterwards to become Engineers, Architects, Builders, Brewers, or Manufacturers (such as Metallurgists, Alkali, Soap, Manure, Glass, or Cement Makers, Bleachers and Dyers, &c.)

Students preparing for the Intermediate Examination in Science and Preliminary Scientific (M.B.) Examination of the University of London should attend the Lectures on Inorganic Chemistry (Winter and Spring Terms).

Candidates for Intermediate Examinations in Medicine will in general require only that part of the course (Summer Term) which relates to Organic Chemistry.

The full course, extending over three terms, will also satisfy the requirements of Students preparing for the Associateship of the Institute of Chemistry, so far as attendance at lectures on General and Theoretical Chemistry is concerned.

1. From October to March (Winter and Spring Terms). About eighty lectures on Inorganic Chemistry and Chemical Philosophy will be given on Mondays, Tuesdays, Wednesdays, and Thursdays from October to December, and on Mondays, Tuesdays, and Wednesdays from January to March, at 9.30 a.m. A Tutorial Class is held in connection with this Course once a week throughout the Session. Fee, £5 5s. for the course.

2. April to June (Summer Term). About thirty lectures will be given on Elementary Organic Chemistry, or the chemistry of the most important series of carbon compounds. This course will include all the subjects required for the Intermediate Examination in Medicine of the University of London. Lecture Days—Monday, Wednesday, and Friday at 12 noon. Fee, £1 11s. 6d.

The General Course (including Inorganic and Organic lectures) qualifies for graduation in the medical faculties of the universities of Edinburgh, Glasgow, Aberdeen, and Durham.

Special Courses of Lectures and of Laboratory Instruction are given for Medical Students preparing for the Conjoint Board Examinations.

Advanced Course.

An Advanced Course for the study of Theoretical Chemistry and those parts of the subject which are required for the degree of B.Sc. in the University of London will meet twice a week. Fee for the session £3 3s.

Laboratory Practice.

The College Laboratory is open daily from 9.30 to 5, except on Saturdays, when it is closed at 1 p.m.

Candidates for Intermediate Examination in Science, Preliminary Scientific (M.B.), B.Sc., and Intermediate Examination in Medicine of the University of London, may obtain in the Laboratory of the College the instruction necessary. The three months Course of Practical Chemistry for the B.Sc., Edinburgh, in the department of Public Health, may be taken in the Mason College Laboratory. Fees:—

	All day.	Three hours per day.
One Term	7 guineas	4½ guineas.
Two Terms	13 „	8½ „
Three Terms	18 „	12 „

A Course of short demonstrations and exercises is given by the Professor or one of his Assistants once a week. All first-year Students are required to attend, unless exempted for special reasons by the Professor. No Fee.

Metallurgy.

Three Courses of Ten Lectures will be given on the Principles and Practice of Metallurgy. Fee, 10s. 6d. for each of the first two courses, and for each of the two sections of the third course. A more advanced course of about sixty lectures upon selected subjects is also given.

There is a separate laboratory for metallurgical students in which provision is made for instruction in assaying, &c.

Evening Classes.

Special Courses of Evening Lectures are arranged during the Winter and Spring Terms of each session. The subjects are treated in a less technical manner and the fees are nominal.

Scholarships.

Priestley Scholarships.—Three Open Scholarships in Chemistry of the value of £100 each are awarded annually in September.

Bowen Scholarship.—One Open Scholarship in Metallurgy of the value of £100 is awarded annually in September.

Forster Research Scholarship.—A Scholarship of the value of £50 is annually awarded.

For particulars apply to the Registrar.

Excursions.

During previous Sessions permission has been obtained to visit some of the great factories in or near Birmingham, in which chemical and metallurgical industries are carried on. Students have thus had most valuable opportunities of gaining a practical acquaintance with some branches of Applied Science. The privilege thus courteously granted by several manufacturers will, it is hoped, be enjoyed in every future Session. The excursions will be conducted by the Professor or Lecturer.

BRADFORD TECHNICAL COLLEGE.

CHEMISTRY AND DYEING DEPARTMENT.

Head Master—W. M. Gardner, F.C.S.

Demonstrator and Lecturer on Geology—A. B. Knaggs, F.C.S.

Lecturer on Botany and Biology—William West, F.L.S.

The school year is divided into three terms. The Session commences on September 14th and terminates on July 17th. The course of instruction extends over two years, and embraces Lecture Courses on Inorganic and Organic Chemistry, the technology of the textile fibres, mordants, natural and artificial colouring matters, technical analysis, and laboratory practice in analytical chemistry, chemical preparations, and dyeing. Fee, £5 per Term, or £13 per Session.

During the first and second terms Evening Classes are held for the benefit of persons engaged during the day and for pharmaceutical students.

ROYAL AGRICULTURAL COLLEGE,
CIRENCESTER.

CHEMICAL DEPARTMENT.

Professor—Prof. E. Kinch, F.C.S., F.I.C.*Assistants*—Cecil C. Duncan, F.I.C., and W. James.

Systematic courses of Lectures are given on the various branches of Chemistry in its relation to Agriculture, illustrated by experiments, and by the collections in the College Museum. They comprise the laws of Chemical Combination and the general Chemistry of mineral bodies, and of the more frequently occurring bodies of organic origin, with the relationships of their leading groups; and, finally, the applications to practical operations of the Chemistry of the atmosphere, of soils and manures, of vegetation and stock feeding, and of the processes and products of the dairy.

In the Laboratory practical instruction is given in the construction and use of apparatus and in Chemical manipulation and analysis, both qualitative and quantitative. After studying the simple operations and the properties of the commonly occurring substances, the Students are taught to analyse a series of compounds, and apply the knowledge thus obtained to the analysis of manures, soils, waters, feeding stuffs, dairy products, and other substances met with in the ordinary course of Agricultural practice. Chemico-agricultural researches are undertaken by the senior Students under the direction of the Professor and his Assistants.

VICTORIA UNIVERSITY.
THE YORKSHIRE COLLEGE, LEEDS.*Professor of Chemistry*—Arthur Smithells, B.Sc. Lond., F.I.C.*Lecturer in Organic Chemistry*—Julius B. Cohen, Ph.D., F.I.C.*Assistant Lecturer and Demonstrator*—Herbert Ingle, F.I.C.*Demonstrator*—T. S. Patterson, Ph.D.

The Session begins October 5, 1897.

Lecture Courses.

1. General Course of Chemistry.—Monday, Wednesday, and Friday, at 11.30 a.m., from October to the end of the second term, and during part of the third term. Fee for the Course, £4 4s.

2. Inorganic Chemistry.—First year Honours Course, Non-metals. Monday, Wednesday, and Friday, at 9.30 a.m. Fee, £3 13s. 6d.

3. Inorganic Chemistry.—Second year Honours Course, Metals. Tuesday, Thursday, and Saturday at 9.30 a.m. Fee, £3 13s. 6d.

4. Organic Chemistry.—Tuesday, Thursday, and Saturday at 12 noon. Fee £3 13s. 6d.

5. Organic Chemistry Honours Course.—Wednesday and Friday at 12 noon. Fee, £2 12s. 6d.

6. Theoretical Chemistry.—Advanced Course. Tuesdays and Thursdays at 9.30 a.m. Fee, £2 12s. 6d.

7. Chemistry as Applied to Coal Mining.—Tuesday during the First Term, at 4 p.m.

8. Chemistry for Teachers.—Saturdays from 9.30 to 12.30 in the first and second terms. Fee, £4 4s.

Laboratory Courses.

The College Laboratory will be open daily from 9 a.m. to 1 p.m., and from 2 to 5 p.m., except on Saturdays, when it will close at 1 p.m.

Fees for the Session—Students working six days per week, £21; five, £18 18s.; four, £16 16s.; three, £13 13s.

Class in Practical Chemistry, Saturday mornings, from 9.30 to 12.30. Fee £1 11s. 6d.

Practical Chemistry for Medical Students.—Tuesdays, 9.30 to 11.30 October to end of December; Thursdays, 2 to 4 from January to end of March.

Practical Course in Sanitary Chemistry.—Tuesdays and Thursdays from 2 to 5 p.m., from January to March. Fee, £5 5s.

Practical Organic Chemistry for Medical Students.—

Tuesdays and Thursdays from 10 to 12 during the Third Term. Fee, £2 2s.

Evening Class.

A Course of twenty Lectures by Mr. Ingle, on the Chemistry of Photography will begin during the first and second Terms. Fee, 10s. 6d.

*Dyeing Department.**Professor*—J. J. Hummel, F.I.C.*Lecturer and Research Assistant*—A. G. Perkin, F.R.S.E.*Assistant Lecturer*—R. B. Brown.

This Course extends over a period of three years, and is intended for those who wish to obtain a full scientific and practical education in the art of dyeing. It is suitable for those who purpose in the future to take any part in the direction of the operations of dyeing or printing of textile fabrics, e.g., the sons of manufacturers, calico printers, managers, master dyers, &c.

*Leather Industries Department.**Professor*—H. R. Procter, F.I.C.

The full Course, which extends over a period of three years, is suitable to all who intend to become Technical Chemists in the Leather Industry, or managers of important works, and is recommended to sons of tanners. The Course includes instruction in chemistry, engineering, leather manufacture, and practical work in the Leather Industries Laboratory.

*Agricultural Department.**Professor*—James Clark, M.A., Ph.D.

The full Course occupies two years, and includes instruction in chemistry, physics, botany, engineering and surveying, and the principles of agriculture, as well as practical work in the various laboratories and out-door agriculture.

Research Students are admitted to the College Laboratories on reduced terms.

Several valuable Scholarships are at the disposal of the College, viz., the Cavendish, Salt, Akroyd, Brown, Emsley, Craven, and Clothworkers' Scholarships, and the Leighton Trustees' Exhibition, and one of the 1851 Exhibition Scholarships. The West Riding County Council Scholarships are tenable at the Yorkshire College.

UNIVERSITY COLLEGE, LIVERPOOL.

Professor—J. Campbell Brown, D.Sc.*Lecturer on Organic Chemistry*—C. A. Kohn, B.Sc., Ph.D.*Lecturer on Metallurgy*—T. L. Bailey, Ph.D.*Demonstrators and Assistant Lecturers*—T. L. Bailey, Ph.D., C. A. Kohn, B.Sc., Ph.D., and A. W. Titherley, M.Sc., Ph.D.*Assistant*—H. H. Froyssell.

The Session commences October 4th.

The William Gossage Chemical Laboratory has been completed and opened for Advanced Students, the Metallurgical Laboratory has been enlarged, and new Gas Analysis and Electro-Chemical rooms have been added, with a third Lecture Room.

The Classes meet the requirements of candidates for the Ordinary B.Sc. Degree, for Chemistry Honours, or for the M.Sc. or D.Sc. Degree in Victoria University; for Degrees in Medicine of Victoria, London, and Edinburgh; for the Pharmaceutical Diplomas; for a special Technological Certificate of University College; and for those studying Chemistry as a preparation for professional, technical, or commercial life. The Classes qualify for the Fellowship of the Institute of Chemistry of Great Britain and Ireland, and other Examination Boards.

Lecture Courses.

General Elementary Course on the principal non-metallic elements and the most important metals, the principles of Chemical Philosophy, and an introductory sketch of Organic Chemistry. Three Terms. Fee, £4.

Engineer's Course of Lectures with Practical Class. Two Terms. Fee, £4.

Dental Course, Lectures and Practical. Fee, £5 5s.
Course A.—Non-metals. Fee, £3 10s.
Course B.—Metals. Fee, £3 10s.
Course C.—Organic Chemistry. Fee, £3 10s.
Course H.—Special Organic Subjects. Fee, £1.
Course D.—Physical Chemistry. One Term. Fee, £1.
Course E.—History of Chemistry and of the Development of Modern Chemical Philosophy. Three Terms. Fee, £2.

Courses F.—Technological Chemistry and Metallurgy: Lectures on Technology are given in connection with Laboratory work at hours to be arranged. The subjects are varied in different years. (1) Alkali and Allied Manufactures. (2) Copper, Iron, and Steel. (3) Lead, Silver and Gold, Aluminium, and other Metals. (4) Distillation of Coal and Tar Industries. (5) Fuel and Gas. (6) Chemistry Applied to Sanitation. (7) Technical Gas Analysis. Three terms. Fee, each course £1 10s.

Practical Classes.

(1) Junior. (2) Intermediate: Qualitative Analysis of Inorganic Substances and of some of the more common Organic Substances. (3) Revision Class. (4) Senior: Practical Organic (Advanced Medical Class). (5) Practical Exercises on Technology, Pharmaceutical Chemistry, Saitanry subjects, Examination of Water and Air, of Animal Secretions, Urinary Deposits, Calculi, and Poisons. (6) Quantitative Class: Course arranged to suit the requirements of the London University B.Sc. Examinations, Pass and Honours, and for Intermediate M.B. Honours.

Chemical Laboratory.

The Chemical Laboratories provide accommodation for every kind of chemical and metallurgical work.

The William Gossage Laboratory, opened in 1897, consists of a large and well-fitted general Laboratory for advanced Students, a new gas analysis room, an additional lecture-room for Metallurgy and other classes, and an addition to the Research Laboratory. New stores for students' apparatus and chemicals have also been built and placed in charge of a skilled dealer.

Students desirous of gaining a thorough theoretical and practical acquaintance with Technical Chemistry, or who intend to adopt Chemical work as a profession, must devote three or four years to special study, for which a full curriculum is provided.

TABLE OF FEES.

Per Week.	One Term, Three Months.	Three Terms, One Session.
One day	£4	£7
Two days	6	10
Three days	8	13
Four days	9	16 10s.
Whole week	10 10s.	21

Pharmaceutical Course (see special syllabus).

Technological Curriculum.

Preliminary Year.—Chemistry, the Elementary Course. Practical Classes 1 and 2. Mathematics, or Mechanics, or Physics. Elementary Engineering, Drawing, and Design (in this or one of the following years). German. Or, the Victoria Preliminary Course and Examination may be taken.

First Year.—Chemistry—Courses A and B; Chemical Laboratory three days per week; Practical Organic Class during the Summer Term; Technological Chemistry, Course F. Physics, with laboratory work, one day per week. Mathematics (intermediate). German. Engineering, First Year Course, Autumn and Lent Terms. Intermediate B.Sc. Examination may be passed.

Second Year.—Chemistry, Lecture Course C, on Organic Chemistry, Lecture Course E or D, Technological Chemistry, Course F, on Metallurgy. Chemical Laboratory, four days per week. Engineering or Physics (Advanced). The Final Examination for the Victoria B.Sc., or the Intermediate Examination of the Institute of Chemistry, may be taken.

Third Year.—Courses D, F, and C. Any other Courses omitted in a previous year. Laboratory, five days per week. Students may finally choose a special subject either of research or of applied Chemistry. The Final Examination for the Associateship of the Institute of Chemistry of Great Britain and Ireland may be taken. Three years study after passing the Preliminary Examination of Victoria University are required for the B.Sc. Degree in the Honours School of Chemistry.

The Sheridan Muspratt Chemical Scholarship of £50 per annum, tenable for two years, will be competed for in December, 1897, on an Examination in subjects which are included in the first two years of the above curriculum. Other Scholarships, Entrance Scholarships, and Free Studentships are also available to Students.

Evening Classes.

Classes, including laboratory work, will be held on Chemistry, Metallurgy, and on Analysis of Gases.

The Prospectus containing full particulars may be obtained from the Registrar, University College, Liverpool.

DURHAM COLLEGE OF SCIENCE, NEWCASTLE-ON-TYNE.

Professor of Chemistry—P. Phillips Bedson, M.A., D.Sc., F.I.C., F.C.S.

Lecturer in Chemistry—Saville Shaw, M.Sc., F.C.S.

Lecturer in Agricultural Chemistry—R. Greig Smith, M.Sc., F.C.S.

Assistant Lecturer and Demonstrator—F. C. Garrett, M.Sc., F.C.S.

The Session will commence on September 27th, 1897.

1. *General Course.*—This Course of Lectures will extend over the three terms of the Session, and is intended to serve as an introduction to the Science. The Lectures will be of an elementary character, and whilst framed to meet the requirements of First Year Students will also be serviceable to such as intend pursuing Chemistry in its various applications in the arts and manufactures, as, for instance, Brewing, Metallurgy, the Manufacture of Soda, Soap, Glass, &c. The subjects treated will include an exposition of the Principles of Chemistry, and a description of the preparation and properties of the chief Elementary Substances, both metallic and non-metallic, and their more important native and artificial compounds. A section of this Course will be devoted to an outline of Organic Chemistry. The class will meet on Mondays, Wednesdays, and Fridays, at 11 a.m., and will commence on Wednesday, October 6th. Fee, £3 10s. for the Session.

2. *Advanced Course.*—Inorganic Chemistry, Tuesdays 3 to 4 p.m., during the Session. Fee, £2; or for students taking Organic Chemistry, £1.

3. *Organic Chemistry.*—A Course of Lectures will be given throughout the Session, the subject of which will be the Chemistry of the Carbon Compounds and an introduction to Chemical Theory. This class will meet on Tuesdays and Thursdays, at 11 a.m., and Fridays 3 to 4 p.m., and will commence on Thursday, October 7th. Fee, £3 10s. for the Session.

Advanced Classes will be formed for the study of Inorganic, Organic, and Theoretical Chemistry. Fee for the course, £3 10s.

A Lecture Course in Analytical Chemistry will be given on Mondays, at 3 p.m.

Metallurgy and Assaying.—Lecturer, Saville Shaw, M.Sc., F.C.S. A Metallurgical Laboratory is provided, in which instruction is given in the ordinary processes of Dry Assaying, and in the preparation and analysis of Alloys, &c. Fee as for Chemical Laboratory.

Agricultural Chemistry.—The instruction in this branch of Chemistry will consist of a series of Lectures and of special practical work in the Chemical Laboratory. Students will be expected to have a knowledge of Elementary Chemistry, such as may be obtained by attending the General Course.

The Lecture Course in Agricultural Chemistry is

arranged for two days a week throughout the Session. Fee, £3 10.

Practical Chemistry.—The Laboratory is open from 10 a.m. to 1 p.m., and from 2 to 5 p.m., except on Saturdays, when it closes at 1 p.m. **Laboratory Fees.**—Students working two days, £2 10s. per term, £6 per session; one day per week, £1 10s. per term, £3 10s. per session.

Courses of Study.—Students will be divided into two classes:—(1) Regular, or Matriculated Students, who are also Members of the University of Durham; and (2) Non-Matriculated Students. Regular Students will be required to follow such a course of study in the subjects professed in the College as will enable them to pass the Examinations for the title of Associate in Physical Science of the University of Durham. Non-Matriculated Students will attend such classes as they may select. Every candidate for admission as a matriculated student must pass an examination on entrance, in reading, writing from dictation, English or Latin Grammar, arithmetic (including decimals), and geography. Registered students in medicine are exempted from this examination, or students who produce a certificate of having passed either of the two following examinations:—

1. Durham Examination for certificate of proficiency in General Education, held in March and September.
2. Durham Examination for Students in Arts in their first year, or any examination of a similar nature that may be accepted by the Council.

Associateship in Physical Science.—Every candidate for the Associateship in Physical Science will be required to satisfy the examiners in—Mathematics, Physics, Chemistry, and either Geology or Natural History—in an examination to be held at the end of the candidate's first year. Associates in Science are admissible one year after obtaining the title of Associate to examination for the degree of Bachelor of Science of the University of Durham.

Exhibitions.—Three Exhibitions of the value of £25, £15, and £10 respectively will be awarded in October next to Candidates desirous of attending the first year course of study in the College.

The examination will be held at the College, and will commence on Wednesday, September 29th.

Evening Lectures.—Courses of Evening Lectures will be given, with a Practical Class for Laboratory instruction.

Two Exhibitions of £15 each will be awarded at the next examination of "Persons not members of the University," which will be held at Durham in March next.

Several other valuable Scholarships are available for students.

OWENS COLLEGE,

VICTORIA UNIVERSITY, MANCHESTER.

Professor and Director of the Chemical Laboratory—Harold B. Dixon, M.A., F.R.S.

Professor of Organic Chemistry—W. H. Perkin, Ph.D., F.R.S.

Demonstrators and Assistant Lecturers—George H. Bailey, D.Sc., Ph.D.; Arthur Harden, M.Sc., Ph.D.; P. J. Hartog, B.Sc.; and E. Haworth, M.Sc.

Lecturer in Technical Organic Chemistry—Jocelyn F. Thorpe, Ph.D.

Assistant Lecturer in Metallurgy—J. Crowther.

The Session begins on October 5, 1897, and ends on July 2, 1898.

The instruction is given by means of Experimental Lectures and Tutorial Classes. The Chemical Classes form part of the Courses for Chemistry in the University.

Chemistry Lecture Courses.

General Chemistry Course.—Tuesdays, Thursdays, and Saturdays, at 9.30, during the two Winter Terms.

Introduction to Organic Chemistry.—Wednesdays and Fridays, at 9.30, during Lent Term.

These courses are intended for Medical Students and others beginning the study of chemistry.

First Year Honours Course.—Mondays, Wednesdays, and Fridays, 11.30 a.m., during the two Winter Terms. The Non-Metals.

Second Year Honours Course.—Mondays, Wednesdays, Fridays, 3.30 p.m., during the two Winter Terms. The Metals.

Third Year Honours Course.—At times to be arranged. Physical Chemistry.

Organic Chemistry (General).—Mondays and Fridays, 9.30, during two Winter Terms.

Organic Chemistry (Advanced).—Tuesdays and Thursdays, 9.30, during the two Winter Terms.

History of Chemistry and Chemical Philosophy.—Wednesdays, 9.30, during the Session.

METALLURGY.—**Lectures:** The Metallurgy of Copper, Lead, Silver, Gold, and the Metallurgy of Iron and Steel will be given in alternate years. **Practical:** The Laboratory will be open to students every day.

The Chemical Laboratories are open daily from 9.30 a.m. to 4.30 p.m., except on Saturdays, when they are closed at 12.30 p.m.

Courses for B.Sc. Degree.—To qualify for the B.Sc. Degree of the Victoria University, Students have to attend a prescribed course of study extending over three years, and to pass the Preliminary Examination of the University either on entering or at the end of a year's Course.

The Honours Course of Chemistry is as follows:—**First year:** First year Honours Lectures; Mathematics (3 hours a week); Physics (3 hours a week); a Language (3 hours a week); Chemical Laboratory (3 days per week). **Second year:** Second year Honours Lectures; General Organic Lectures; Applied Chemistry Lectures; Physics Laboratory (1 day per week); Chemical Laboratory (3 days per week). **Third year:** Third year Honours Lectures; Honours Organic Lectures; History of Chemistry Lectures; Chemical Laboratory (5 days per week).

The following awards are made to successful Students in the Honours Examination:—A University Scholarship of £50; a Mercer Scholarship of £25. A University Fellowship of £150 is awarded annually among the Graduates in Science for the encouragement of Research. Among the College Scholarships open to Chemical Students are the Dalton Chemical Scholarship, £50 per annum for two years; the 1851 Exhibition Scholarship; the John Buckley Scholarship; &c.

Applied Chemistry.

First Course.—Sulphuric Acid and Alkali Manufactures. General Principles of Chemical Engineering.

Second Course.—The Chemistry of Fuel. The Manufacture of Illuminating Gas and Gaseous Fuel.

Third Course.—Natural and Artificial Dye-stuffs, and the Principles of Dyeing and Printing.

Certificates in Applied Chemistry.

The course extends over a period of three years, and comprises systematic instruction by means of lectures and practical work in the laboratories.

Before admission to the first year's course students are required to give such evidence of elementary knowledge of Mathematics and Chemistry as shall be considered satisfactory by the Senate.

The first year's course is the same for all students working for the certificate.

In the second and third years a choice may be made between Inorganic and Organic Chemistry. By this division of the subject a student wishing to apply himself specially to the inorganic side of the science, may attend during his second year the Honours course in Metals, and courses on Geology or Mineralogy, and during his third year, courses on Metallurgy and on Geology or Mineralogy; while a student wishing to apply himself specially to the organic side of the science, may attend during his second and third years the Courses on Organic Chemistry, and courses on the Coal Tar Colours and on Dyeing and Printing.

Part of the Laboratory practice in the second and third years will consist in the examination and analysis of raw materials, products from chemical works, &c., in connection

with the special courses of lectures on Applied Chemistry. In the Chemistry and Physical laboratories the practical work in the second year will be arranged in accordance with the branch of Chemistry selected by the candidate.

In the third year the student, if sufficiently advanced, will be set to work on some analytical process or problem in Applied Chemistry, under the direction of the teaching staff.

UNIVERSITY COLLEGE, NOTTINGHAM.

DEPARTMENTS OF CHEMISTRY AND METALLURGY.

Professor of Chemistry—Frank Clowes, D.Sc. Lond., F.I.C.

Demonstrators of Chemistry—J. J. Sudborough, D.Sc., Ph.D., F.I.C.; R. M. Caven, B.Sc., F.I.C.; and G. Mel-land, B.Sc., A.R.S.M., F.I.C.

The Classes of the College are open to students of both sexes above sixteen years of age.

The Session commences on October 11th.

Lecture Courses.—The Chemistry Day Lectures extend over three years. In the first year a student enters for the course on Non-Metals for the first two terms and for Elementary Organic Chemistry in the third term. In his second year he takes the course on Metals for the first two terms. In his third year he attends a course on Advanced Organic Chemistry or Applied Chemistry. Fee for Day Lectures and Classes: Non-Metals or Metals 42s.; Organic Chemistry (one term) 21s.; Advanced Organic Chemistry, 21s. per term.

Demonstrations and Lectures on Analytical Chemistry will be given in the day and evening, and should be attended by all students.

A Chemical Calculation Class is also held. Fee per Term, 5s.

Students may qualify themselves by attendance at these lectures and classes for the Examinations of the Universities of London, Cambridge, or Oxford, and for the Medical Examinations of the Royal College of Surgeons and of the Universities of Cambridge and Edinburgh: they may also obtain instruction in Chemistry for technical or other purposes, and can enter for a full Chemical Engineering Curriculum. Special attention is given to the requirements of candidates for the Associateship of the Institute of Chemistry.

Practical Chemistry and Metallurgy.—The Chemical and Metallurgical laboratories are open every day from 9 to 5, except on Saturday, when the hours are from 9 to 1; also on Tuesday and Thursday evenings from 7 to 9. Each Student works independently of other Students at a course recommended by the Professor. Instruction is given in general Chemical Manipulation, in Qualitative and Quantitative Analysis, and in the methods of Original Chemical Investigation and Research; Students are also enabled to work out the applications of Chemistry to Pharmacy, Metallurgy, Dyeing, Agriculture, Brewing, Iron and Steel, Tanning, and other Manufacturing Processes. Fees for day students: For one term, £7; for the session, £18; for six hours weekly 40s., and 5s. extra for each additional hour per week. For evening students, 10s. for two hours per week, three hours 15s., four hours 20s., six hours 30s., per term.

Courses of Technical Chemistry Lectures are also given on Engineering, Dyeing and Bleaching, Brewing, Plumbing, Bread-making, Gas Manufacture, and on other processes of applied Chemistry.

Pharmaceutical Students can at all times work in the Chemical Laboratory, taking work suitable for the preparation for the Minor Examinations. Special lectures will also be given in Chemistry and Materia Medica.

Government Lectures and Classes.—Evening Lectures and Laboratory instruction will be given by the Demonstrators of Chemistry to Students who intend to present themselves for Examination by the Government Science and Art Department in May next. Inorganic, organic, and practical chemistry, agricultural chemistry, and metallurgy will be taught in the elementary, advanced, and

honours stages, each of which commences at the beginning of the College Session in September. Fee for each Lecture Course, 5s.; for each Laboratory Course, 10s.

An Agricultural Course of instruction, extending over two years, is now organised under the general direction of Mr. M. J. R. Dunstan, M.A., F.R.S.E. It includes instruction in chemistry, botany, agriculture, with practical work on experimental fields, dairy work, farriery, land surveying, &c. The instruction is designed for those who intend to become farmers, bailiffs, land agents, or colonists, and may be extended to a third year if desired. Fee, £15 per annum for residents in Notts, £20 to residents in other counties.

Full information concerning all College Classes is given in the College Prospectus, price one penny.

UNIVERSITY COLLEGE, SHEFFIELD.

Professor of Chemistry—W. Carleton Williams, B.Sc., F.C.S.

Demonstrators and Assistant Lecturers—G. Young, Ph.D., and L. T. O'Shea, B.Sc., F.C.S.,
The Session will commence on October 6th.

First Course.—Chemistry of the Non-Metallic Elements. Tuesday and Friday from 10 to 11 a.m. Fee, £2 12s. 6d.

Second Course.—Chemistry of Metals. Monday and Thursday from 10 to 11 a.m. £2 12s. 6d.

Third Course.—Inorganic Chemistry, Honours Course, Monday, 3. Fee, £1 11s. 6d.

Organic Chemistry.—Elementary: Wednesdays, 10 to 11; fee, 15s. Honours: Tuesdays and Thursdays, 12 to 1; fee, £2 12s. 6d. Advanced: Thursdays, 4 to 5; fee, £1 11s. 6d. Special Course: Hours to be arranged; £1 1s. Chemistry of the Colouring Matters: Fridays, 12 to 1; fee, £1 11s. 6d.

Short Courses of Lectures are also given by L. T. O'Shea on the Chemistry of Coal Mining, and on Thermic Chemistry.

A Course of Lectures is arranged for Medical Students, with a special class in Qualitative Analysis.

Laboratory.—Working hours to be arranged between Professor and Students.

Sessional Fees for Day Students:—Six hours per week, £5 5s.; Nine, £7; Twelve, £8 8s.; Eighteen, £11 5s.; Twenty-four, £14; Thirty-two, £17.

Day Students may not enter for less than six hours a week. Students joining the Laboratory at Christmas will be charged two-thirds and at Easter one-third of the Fees for the whole Session.

Fees for short periods (working thirty-two hours per week):—For one month, £3 3s.; two months, £5 5s.

An arrangement has been entered into with the Science and Art Department, South Kensington, which will enable Science Teachers to work in the Chemical Laboratory for three, six, or twelve hours a week on payment of one-quarter of the usual fee, the Department being willing to pay the remainder under certain conditions, of which full information may be obtained on application to the Registrar.

Evening Classes.—Lectures, Wednesday, 8 to 9. Laboratory instruction, Wednesday, 6 to 9, and another series to be arranged if desired. Sessional Fee, one evening per week, £1 10s.; two, 50s.; or Lecture Class and Laboratory, on Wednesday evening, £1 10s. Fee for one term, 17s. 6d.

UNIVERSITY COLLEGE, DUNDEE.

UNIVERSITY OF ST. ANDREWS.

Professor of Chemistry—James Walker, Ph.D., D.Sc.

Assistant Lecturer—J. S. Lumsden, Ph.D., B.Sc.

Lecture Assistant and Laboratory Steward—J. Foggie, F.C.S.

The Winter Session begins on October 6th, and ends on March 16th. The Summer Session extends from the middle of April to the end of June.

The First Year's Lecture Course on Systematic Chemistry is given daily during the Winter Session, and

embraces the Elements of Inorganic and of Organic Chemistry.

Advanced Courses, of about fifty lectures each, will be given during the year as follows:—

Organic Chemistry; Inorganic Chemistry, including the more important technological applications; Theoretical and Physical Chemistry; Bleaching and Dyeing, including the Chemistry of the Textile Fibres.

Practical Instruction in all of the above branches will be given in the Laboratories and Dye-house. Special facilities are afforded to Research Students.

The Lectures and Laboratory Practice in Chemistry are recognised by the Medical Colleges of London and Edinburgh. The Courses are suitable for the degrees of the University of London and for the Civil Service appointments, and will also satisfy the requirements of Students in Pharmacy, and of Students who intend to become candidates for the Associateship of the Institute of Chemistry, as far as qualification in Chemistry is concerned.

UNIVERSITY OF EDINBURGH.

DEPARTMENT OF CHEMISTRY.

Professor—Alex. Crum Brown, M.D., D.Sc., F.R.S.

Lecturers—L. Dobbin, Ph.D., and H. Marshall, D.Sc.

Assistants—W. W. Taylor, M.A., B.Sc., and J. P. Longstaff.

The working terms are—Winter Session, from middle of October to middle of March; Summer Session, from beginning of May to end of July.

Lecture Courses.—During the Winter Session a General Course of Chemistry for medical and science students is given by the Professor. The class meets daily; fee £4 4s. An Advanced Course of twenty-five lectures is also given in the Winter Session; fee, £2 2s. A class on Organic Chemistry is held in summer; fee, £2 2s. There is also a class on Chemical Theory, by Dr. Dobbin; fee £1 1s.; and a class on Mineralogy and Crystallography, by Dr. Marshall; fee, £2 2s. All these Lectures, except the General Course, are now open to women.

In addition to the above, Lecture Courses are given by the Assistants on some particular branch of Organic and Inorganic Chemistry. These Lectures are free to Laboratory Students.

Tutorial classes are held in connection with the General Course.

Laboratories.—Practical classes for Medical Students meet daily during the latter part of the Winter Session and in the Summer Session. (Fee, £3 3s.) The laboratories for analytical and advanced practical work are open daily from 9.30 till 4.30. (Fees: Whole Day—Winter Session, £10 10s., Oct.-Dec., Jan.-March; or Summer Session, £5 5s. Half Day—Winter Session, £5 6s., Oct.-Dec., Jan.-March; or Summer Session, £3 3s. Preference will be given to students in the above order. Students who are not Matriculated may attend the Chemical Laboratory on payment of the entrance fee of 5s. in addition to the Laboratory fees. Full Courses of instruction are given in Analytical, Practical Organic and Inorganic Chemistry, including Gas Analysis, Metallurgy, and Assaying. Facilities are afforded to advanced students who desire to undertake chemical investigations.

Various prizes and scholarships are attached to the laboratory and general class.

Graduation.—Two Degrees in Pure Science are conferred, viz., Bachelor of Science (B.Sc.) and Doctor of Science (D.Sc.).

Candidates for Degrees in Science, if not graduates (by examination) in Arts in one of the Universities of the United Kingdom or in a Colonial or Foreign University recognised for the purpose by the University Court, must pass a preliminary examination in (1) English; (2) Latin, Greek, French, or German; (3) Mathematics; (4) One of the languages Latin, Greek, French, German, Italian, not already taken under (2), or Dynamics. In the case of a student whose native language is other than European, the Senatus may, at the Preliminary Examination, accept

such language as a substitute for a modern European language. The Senatus may also in such a case accept as an alternative to Latin or Greek any other classical languages, such as Sanscrit or Arabic.

The First B.Sc. Examination embraces Mathematics, or Biology (*i.e.*, Zoology and Botany), Natural Philosophy, and Chemistry. The Second B.Sc. Examination includes any three or more of the following subjects:—Mathematics, Natural Philosophy, Astronomy, Chemistry, Human Anatomy, including Anthropology, Physiology, Geology, including Mineralogy, Zoology, including Comparative Anatomy, and Botany, including Vegetable Physiology. Chemistry in this examination embraces Inorganic, including Mineralogical, Chemistry; Organic Chemistry; Physical Chemistry; Chemical Crystallography; History of Chemistry. Practical Examination:—Complex Qualitative Analysis; Quantitative Analysis, including Gas Analysis and Organic Analysis; Preparation of Pure Substances, organic and inorganic; Physico-chemical Measurements.

In the Courses for Final Examination in Pure Science, two written papers are set in each subject professed, the second of a higher standard than the first. Candidates must pass the first section in all, and the second section in at least one, of the subjects professed; the same regulations apply also to the Practical and Oral Examinations.

A candidate for the D.Sc. Degree must submit a thesis on original work done by him. The Thesis must be approved before the candidate is allowed to proceed to Examination. The candidate in Chemistry may be required to pass a searching examination in one of the following branches:—(1) The Chemistry and Chemical Technology of Inorganic Bodies, including Metallurgy; (2) Organic Chemistry; and to show a thorough practical acquaintance with chemical analysis in all its branches, and with the preparation of pure substances.

HERIOT-WATT COLLEGE, EDINBURGH.

Professor—John Gibson, Ph.D., F.R.S.E.

Assistant Professor—(Vacant).

Demonstrators—Andrew F. King and James B. Shand. The Session begins October 5th, 1897.

The curriculum of this College comprises both Day and Evening Classes, each department providing the higher general and technical education.

Chemistry.—The first course for day students is a combination of Lectures with Laboratory instruction. In the Lectures some of the more important elements and their compounds are discussed in detail, so as to lead to a knowledge of the general laws of chemical action. Other important elements are treated in less detail, and the relations and classification of the elements generally are broadly indicated. In the Laboratory each student will receive instruction in general chemical manipulation, in accurate weighing, volumetric measurements, and in some of the simpler methods of quantitative analysis. After making a series of simple preparations, he works through a number of experimental exercises illustrating chemical combination, oxidation, reduction, and double decomposition. These exercises are followed by instruction in simple methods of qualitative analysis, especial attention being given to dry way testing and the use of the spectroscope. Students attending a further course may take up the study of systematic analysis, and extend the knowledge they have gained of quantitative analysis by exercises in gravimetric, volumetric, and electrolytic methods. Ultimately they may make a speciality of any branch of the subject which may be most necessary for their future work. Great attention has been paid to the thorough equipment of Advanced Laboratories, and special facilities are given to advanced students who may wish to engage in any class of Research (Inorganic or Organic) whether of a purely chemical or of a technical nature.

The teaching in the Evening Classes is based on the Syllabus of the Science and Art Department, and in-

cludes Elementary, Advanced, and Honours Courses in Theoretical and Practical Inorganic and Organic Chemistry.

GLASGOW AND WEST OF SCOTLAND TECHNICAL COLLEGE.

Professor of Chemistry—G. G. Henderson, D.Sc., M.A.
Professor of Technical Chemistry—E. J. Mills, D.Sc., F.R.S.

Agricultural Chemistry Lecturer—John W. Paterson, B.Sc., Ph.D.

Professor of Metallurgy—A. Humboldt Sexton, F.C.S., F.R.S.E.

The main objects of this College are to afford a suitable education to those who wish to qualify themselves for following an industrial profession or trade, and to train teachers for technical schools. It was founded by an Order in Council, dated 26th November, 1886, according to a scheme framed by the Commissioners appointed under the provisions of the Educational Endowments (Scotland) Act, whereby Anderson's College, the Young Chair of Technical Chemistry in connection with Anderson's College, the College of Science and Arts, Allan's Glen's Institution, and the Atkinson Institution were placed under the management of one governing body.

The Diploma of the College is awarded to Day Students who have attended prescribed courses of instruction and passed the necessary examinations. The ordinary courses extend over three years, but arrangements are made for advanced students continuing their studies in special departments.

Complete courses of instruction in Metallurgy and Mining will be given in both Day and Evening Classes.

Copies of the Calendar for 1896-97 may be had from Mr. John Young, B.Sc., the Secretary, 38, Bath Street, Glasgow, price by post, rs. 4d.

UNIVERSITY OF ST. ANDREWS.

UNITED COLLEGE OF ST. LEONARD AND ST. SALVATOR.

Professor of Chemistry—T. Purdie, B.Sc., Ph.D., F.R.S.

The Session begins on October 6th. A Competitive Examination, open to intending Students of Arts or Science, for about fifty Entrance Bursaries, ranging in value from £40 to £10 each per annum, will be held on September 25th and following days. About thirty of these Bursaries are restricted to Men and twenty to Women, the latter being intended for women who at the conclusion of their Arts or Science Course will proceed to Medicine. Two are open to students of either sex. Two Scholarships of £100 each, tenable for one year, will be open for competition to Graduates of Science at the close of Session 1897-98. A Hall of Residence is provided for Women Students. Two Degrees in Science are conferred by the University of St. Andrews, viz., Bachelor of Science (B.Sc.) and Doctor of Science (D.Sc.), and Chemistry is also included in the curriculum for the M.A. Degree; the regulations will be found in the "University Calendar."

Lecture Courses.

Two distinct Courses of Lectures are given, each comprising at least one hundred meetings of the class.

First Year's Course.—This Class meets at 11 o'clock on five days in the week. The introductory lectures treat of the Nature of Chemical Action, the Classification of Substances into Elements and Compounds, the Phenomena of Oxidation, and the Composition of Air and Water. The Laws of Chemical Combination and the Atomic Theory are next discussed, after which the more commonly occurring elements and inorganic compounds are described systematically. Elementary Organic Chemistry is also included in the Course.

The chemistry of manufactures is referred to only cursorily; special attention, on the other hand, is

given to those parts of the science which are of general educational value, and as much of the theory of chemistry is introduced as is compatible with elementary treatment. The Lectures are supplemented by a short Course of Laboratory Practice, intended to illustrate the principles of the science.

These courses of instruction are intended to meet the requirements of the Arts' Curriculum; also of candidates for the First B.Sc. Examination, and of students of medicine, so far as Theoretical Chemistry is concerned.

Second Year's Course.—The first part of the Course is devoted to Organic Chemistry, and the second part treats of the General Principles and Theory of Chemistry, and of more advanced Inorganic Chemistry, the instruction in general being such as is required for the Second B.Sc. Examination.

Certificates are awarded on the results of examinations, and the "Forrester Prize" of about £10 is awarded to the best Student of the year.

Fee for the Session, for each Course, £3 3s.

Practical Chemistry.

The Laboratory is open daily from 9 a.m. to 4 p.m., except on Saturdays, when it is closed at 1 p.m. The work pursued in the Laboratory comprises:—(1) The performance of experiments illustrative of the Principles of Inorganic and Organic Chemistry; (2) Qualitative and Quantitative Analysis; (3) Original Investigation. Each student pursues an independent course of study under the supervision of the Professor or Demonstrator, the nature of the work varying with the proficiency of the student and the particular object he may have in view. Suitable courses of instruction in Practical Chemistry are provided for candidates for the First and Second B.Sc. Examinations, and for Students of Medicine.

The fees for Practical Chemistry vary according to the number of hours taken weekly. A certain number of working places in the Laboratory will be available without fee for students who are capable of undertaking original investigation.

QUEEN'S COLLEGE, BELFAST.

Professor—E. A. Letts, Ph.D., D.Sc., F.R.S.E., &c.

The Session commences on Tuesday, October 19, 1897.

I.—Chemistry.—The lectures are delivered at 3 p.m., on the first five days of each week until the beginning of April, and on three days of each week after May 1st, at 2 p.m. The course is divided into three parts:—(1) Chemical Philosophy; (2) Inorganic Chemistry; (3) Organic Chemistry. Fee, £2.

II.—Practical Chemistry.—In this course the Students are instructed in the general methods of conducting Chemical Analyses. Fee, £3.

III.—Laboratory Pupils.—The Chemical Laboratory is open from November until the end of March, and from May 1st until the third week of July, on the first five days of the week, from 10 a.m. until 4 p.m. Students are admitted as working pupils on payment of a fee of £5 for the first period, or of £3 10s. for the second period (or for a single term).

Scholarships.—In addition to various Scholarships awarded in the Faculties of Arts and Medicine in which Chemistry forms a part of the examination, there are other valuable Scholarships awarded specially in connection with the schools of Chemistry and Physics.

QUEEN'S COLLEGE, CORK.

Professor—Augustus Edward Dixon, M.D.

Demonstrator—R. E. Doran, F.C.S.

The College Session begins on October 19th, 1897, and ends on June 11th, 1898. The classes are open to male and female students.

Systematic Chemistry.—(1) General course of Inorganic Chemistry, Elementary Organic Chemistry, and Chemical Philosophy.—Fee for each Sessional Course, £2. Each

subsequent Course, £1. (2) Advanced Organic Chemistry, and Chemical Philosophy.

Practical Chemistry.—(1) Two ordinary Courses of Practical Chemistry will be held, each of three months' duration; one commencing on January 3rd, 1898, and adapted to the requirements of Students proceeding to the Examinations of the Royal University of Ireland; the other ending about the last week in June, and suitable for Medical Students intending to present themselves for the Examinations of other Licensing Bodies. Fee for each Sessional Course, £3. (2) A Course for Pharmaceutical Students will be held in the second and third terms; fee, £5. (3) Special Courses.

The Chemical Laboratory is open daily from 10 to 4 o'clock (except during class hours and on Saturdays) under the Superintendence of the Professor, to Students entering for special courses of qualitative and quantitative analysis; organic chemistry; or for the purpose of original investigation.

QUEEN'S COLLEGE, GALWAY.

Professor—Alfred Senier, Ph.D., M.D., F.I.C.

Demonstrator—Hugh Ryan, B.A.

The College Session is divided into three terms. The First Term extends from October 19 to December 22, the Second Term from January 6 to April 2, and the Third Term from April 18 to June 11.

Chemistry is studied by attendance at Lectures, by work in the Laboratories, and by the use of the College Library. The Courses in the several faculties are arranged with a view to the requirements of the Royal University of Ireland, but are adapted also to those of other Universities and licensing bodies.

Lecture Courses. Faculty of Arts.—1. Second year's Course, Inorganic and the Elements of General Chemistry. 2. Third year's Course, Advanced Organic Chemistry. 3. Fourth year's Post-Graduate Course, Advanced General Inorganic and Organic Chemistry. *Faculty of Medicine.*—First year's Course, Inorganic and Elementary Organic Chemistry. *School of Engineering.*—First year's Course, Inorganic Chemistry.

Laboratory Courses. Faculty of Arts.—1. Second year's Course, Exercises in Inorganic Qualitative Analysis. 2. Third year's Course, Quantitative Analysis and other experiments to suit the requirements of individual Students. 3. Fourth year's Post-Graduate Course, Advanced Quantitative Analysis, Organic and Inorganic Preparations, and determination of their Physical and Chemical characters. 4. The Laboratories are also open to Students for work in other branches of Chemistry. *Faculty of Medicine.*—1. Second year's Course, Inorganic and Organic Elementary Qualitative Analysis, and the Chemical Examination of Urine. *School of Engineering.*—1. Second year's Course, Inorganic Qualitative Analysis.

For Fees, Regulations as to Scholarships, and other particulars apply to the Registrar, from whom the Calendar, published in December, and the Extracts from Calendar, published in advance in July, may be obtained.

ROYAL COLLEGE OF SCIENCE FOR IRELAND, STEPHEN'S GREEN, DUBLIN. (SCIENCE AND ART DEPARTMENT).

Professor of Chemistry—W. N. Hartley, F.R.S.

Assistant Chemist—Hugh Ramage, F.I.C., Associate of the Royal College of Science, Dublin.

Demonstrator of Chemistry and Assaying—J. Holms Pollok, B.Sc.

The Session commences on Tuesday, October 5th, 1897.

The Royal College of Science for Ireland supplies, as far as practicable, a complete course of instruction in Science applicable to the Industrial Arts, and is intended also to aid in the instruction of teachers for the local Schools of Science.

Diplomas are awarded in the Faculties of Mining, Engineering, and Manufactures, Physics, and Natural Science. The Diploma of Associate of the Royal College

of Science in the Faculty of Manufactures is recognised by the Council of the Institute of Chemistry of Great Britain and Ireland as qualifying candidates for admission to the practical examinations of the Institute.

The instruction in Chemical Science includes (1) General Chemistry; (2) Advanced Chemistry, including Chemical Manufactures and Metallurgy; (3) Analytical and Experimental Chemistry; (4) Instructions in Chemical Research.

Fees payable by Non-Associates:—£2 for each separate Course of Lectures. For Analytical Chemistry and Research—£2 for a special course of one month; £5 for three months; £9 for six months; £12 for the entire session. For Assaying—£5 for three months; £9 for six months. £12 for the entire session.

NOTE.—Important changes have been made in the Curriculum for Associate Students. Full particulars are contained in the Directory of the College, which may be had on application to the Secretary.

The following are supplementary courses of instruction arranged for those who are attending a Course of Lectures:—

- (1) Laboratory Instruction in the Theory of Chemistry.
- (2) An Analytical Course for Students in Engineering.
- (3) A Course of Practical Chemistry for Medical Students.
- (4) The Analysis of Water, Air, Food, and Drugs, intended for the instruction of Public Analysts and Medical Officers of Health.
- (5) Assaying.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1973.

VANADIUM IN RUTILE.

By W. B. GILES, F.I.C.

IN the CHEMICAL NEWS (vol. lxxvi., p. 102) there appears a paper by B. Hasselberg entitled "Notes on the Chemical Composition of the Mineral Rutile," in which the following passage occurs:—"While the presence of vanadium in the rutile thus forms a *hitherto entirely unknown feature of this mineral*." I wish to point out that this is not correct, for rutile has been known to contain vanadium for over thirty years. Ste.-Claire Deville showed that this mineral contained not only vanadium, but also molybdenum. He says indeed in his paper, "Ou voit que le rutile est une matière dont on peut extraire le vanadium avec le plus grand avantage." 11

In 100 grms. of the rutile from Saint Yrieix he found—

0.323 grm. of vanadic acid, and
0.486 grm. of molybdic acid.

He also shows that vanadium is found in bauxite, in most clays, in cerite from Bastnäs, and in cryolite, as well as in the emerald and a variety of other substances. I may further point out that in these interesting researches he established the presence of tellurium and titanium, as well as vanadium in cerite, of tantalic acid in wolfram, and of niobic acid in cryolite. The methods he employed for the detection and estimation of the vanadium are very good, and probably no better way of effecting the separation of this widely-diffused element is known even at the present time. These researches are to be found in the *Annales de Chimie et de Physique*, 3me Series, t. lxi., and also in the *Comptes Rendus*, t. xlix., pp. 210 to 301.

ON A QUANTITATIVE SEPARATION OF ARSENIC FROM ANTIMONY.

By OSCAR PILOTY and ALFRED STOCK.

THE quantitative separation of arsenic, antimony, and tin is one of the most difficult problems of mineral analysis if the separation of each element is required.

We shall now describe a procedure which we have elaborated for the separation of arsenic from antimony, and which seems to us calculated to solve this question with accuracy and expedition. We have carried out this method quantitatively as regards arsenic and antimony. The separation from tin has been tried only qualitatively.

The observation upon which our method depends is the volatility of arsenic with hydrogen sulphide in a strongly hydrochloric acid solution. This fact may perhaps throw a light on the causes of some of the many discrepancies observed in the precipitation of arsenic as sulphide. If we heat arsenic tersulphide with very strong hydrochloric acid until there is a brisk discharge of hydrochloric acid, the chief part of the arsenic escapes with moderate ease, and the yellow arsenic sulphide disappears almost entirely. Indeed our experiments showed that from a solution of arsenic teroxide or pentoxide no sulphide is precipitated by sulphuretted hydrogen if it is heated to ebullition with a simultaneous introduction of gaseous hydrochloric acid. Under these conditions the arsenic distils away entirely in a short time from the solution, probably as trichloride.

From the foregoing it will be readily seen that the precipitations of arsenic can be accurate only if the solutions

containing the metal are but slightly acid, or are not heated in presence of much hydrochloric acid.

We must emphasize this condition the more as we cannot find it mentioned either in journalistic literature or in standard works on chemical analysis. On the contrary, it has often been definitely shown that arsenic pentasulphide precipitates in heat from a hydrochloric solution, and contains tersulphide in abundance, in full contradiction with the statements of Bunsen concerning the precipitation of pentasulphide. Our observation seems to us fully to explain this contradiction.

The partial reduction of arsenic acid on heating in concentrated hydrochloric acid has been already noticed. Lately Neher has reported on this observation. He informs us that under certain circumstances, on precipitating such a boiling solution with hydrogen sulphide, there is formed above the liquid a cloud of arsenic trisulphide. But he also failed to notice the behaviour of concentrated hydrochloric solution on the action of sulphuretted hydrogen, and the decomposability of the tersulphide already precipitated.

We should here remark that the precipitation of the arsenic in the slightly hot hydrochloric solution, as first used by Bunsen, always gives excellent results. The attributes of arsenic pentasulphide are so striking that the weighing of arsenic in this form seems preferable to all other methods.

Since other metals on the permanent presence of an excess of hydrochloric acid are not affected by hydrogen sulphide, it was our task to apply the described behaviour to arsenic for its quantitative separation from all other metals. We undertook, in the first place, its separation from antimony, and we obtained very accurate results.

We are now engaged with experiments to dispense with a double precipitation of arsenic.

We have hitherto practically effected only the separation of arsenic from antimony. We shall next report on the separation of arsenic from tin and the other elements of arsenic sulphide group.—*Berichte*, No. 12, p. 164.

ON THE PURIFICATION AND ATOMIC WEIGHT OF CERIUM.*

By MM. WYROUBOFF and VERNEUIL.

OF all the metals which we are in the habit of calling rare, cerium is without doubt the least known. A large number of researches have been devoted to it, and one would think in reading them that the subject must be well nigh exhausted. However, in taking the facts which seem the most incontestable one by one, we soon see that we are in a domain of uncertainty and contradictions. We only know the atomic weight of cerium approximately, we are uncertain as to its valence, and we are not even quite sure of its identity. Is it really a simple element, as we have hitherto attempted to believe, or does it consist of a group like the didymium of Mosander? like the erbium of Bahr and Bunsen? This last opinion has been upheld quite recently by the late M. Schützenberger in a series of important memoirs (*Comptes Rendus*, cxx., pp. 663, 962; cxxiv., p. 481). There should be, according to this eminent chemist, several elements presenting all the chemical and physical characteristics of cerium, and having atomic weights varying from 85 to 104 (Ce being taken as bi-valent). If such were the case, all the chemistry of cerium which has been done up to the present would have no longer any *raison d'être*, and every effort should be directed towards the separation of the different simple bodies of which it is formed.

We now extract, from a comprehensive research which will appear shortly, that which appears to concern the

* *Bull. Soc. Chim.*, Series 3, vol. xvii.-xviii., No. 14.

capital question of the identity of cerium, for the purpose of showing that it is a definite, distinct body, possessing always—no matter what its origin may be—the same atomic weight, and incapable of being split up into more simple elements by any known process.

Two misconceptions dominate the whole chemistry of cerium; the one has regard to its separation from its neighbouring metals, the other to the determination of its atomic weight. It is these two misconceptions which appeared to us important to first of all clear up.

Purification of Cerium.—There are three impurities associated with cerium which are very difficult to get rid of. These are, in order of difficulty of elimination, iron, didymium-lanthanum-yttria, and thoria.

Iron.—It is generally believed that a precipitation of oxalic acid, or oxalate of ammonia, suffices to completely eliminate the iron. This is entirely erroneous. Two or even three precipitations, in warm and acid solution, hardly suffice to remove the last traces of iron. Its presence, even in minimal quantities, is manifested by the colour of the calcined ceroso-ceric oxide, which takes a more or less pronounced rose tint, sometimes even reddish.

Didymium and Lanthanum.—We have admitted, as a fact without question, that Debray's process—which consists, as is well known, in the twice-repeated fusion with nitre at about 320° —separates integrally the cerium from the other metals of its group. Nothing, however, is less certain. This process, which is purely empiric, appears to be based on the more and more difficult decomposition of the nitrates of protoxide of cerium, didymium, and lanthanum; in reality it is accompanied by a phenomenon infinitely more complex. The cerous nitrate passes firstly to the state of ceric nitrate, which easily gives an extremely stable basic salt, and this nitrate combines at a higher temperature with the nitrates of didymium and lanthanum to form a complex oxidised salt, to which we shall return later. We can follow this reaction by evaporating the red solution obtained by dissolving the calcined oxides in nitric acid to dryness, and heating to increasing temperatures. Towards 120° , when all the nitric acid is driven off, the mixture has a bright yellow colour. If at this moment we treat it with water, we obtain a pale yellow insoluble body, consisting of absolutely pure cerium in the state of nitrate $(\text{Ce}_2\text{O}_4)_4 \cdot \text{N}_2\text{O}_5$, and a violet liquid containing cerium in the state of protoxide, with all the lanthanum and didymium. On reheating the mass, it gives off nitrous fumes and takes a chamois colour, which becomes accentuated as the heating proceeds; taken up with water, the mass gives an opalescent liquid which it is impossible to filter. This change of colour and of properties indicates that a new nitrate, of an oxide altogether different to Ce_2O_4 , is formed. In fact, in the presence of protoxides of more energetic basicity, the ceroso-ceric oxide tends to form an oxide, $\text{Ce}_3\text{O}_4 \cdot 3\text{MO}$, which becomes remarkably stable at high temperatures when $\text{M} = \text{Di}$ or La . This oxide, which we propose to study in detail in a future note, gives very beautiful salts, which do not by any means resemble the yellow salts of the oxide Ce_2O_4 . It follows from this that Debray's process does exactly the opposite to what he proposes; instead of separating, it tends to combine Ce_3O_4 with $\text{DiO} + \text{LaO}$. No doubt by keeping the temperature up to 330° for a long time we can decompose this complex nitrate, but this decomposition is only achieved with great difficulty; it is for this reason that we can only arrive at a more or less complete separation, after a long series of fusions. It appeared to us to be much more rational to stop the reaction at the moment when the oxide Ce_3O_4 cannot combine with the $\text{DiO} + \text{LaO}$, for at this moment the cerium should be absolutely free, not only from the two other metals, but also from the metals of the yttria group which are always found in company with didymium.

Experience fully confirms this forecast, and we have been able to prepare in one day several hundred grms. of

perfectly pure cerium. This is how we set to work:—We calcine the oxalates gently, and treat them with nitric acid; here two different cases may present themselves.*

a. If the mixture contains more than 50 per cent of cerium, nitric acid will not dissolve it integrally, even with the aid of heat. One might be inclined to believe that the insoluble residue is formed of the pure oxide Ce_2O_4 , but such is not the case. It is a complex combination of the oxide Ce_3O_4 with $\text{DiO} + \text{LaO}$. In this case it is necessary to dissolve the oxalates in nitric acid, add an excess of peroxide of hydrogen and ammonia, and boil. The voluminous reddish brown precipitate of peroxide of cerium, and of the peroxides of Di and La which are formed, quickly lose oxygen, and become first orange-colour and then yellow. Having arrived at this state they then consist of ceroso-ceric hydroxide, $\text{Ce}_3\text{O}_4 \cdot 3\text{H}_2\text{O}$, mixed with the protoxides of DiO and LaO . It only remains now to wash the precipitate to get rid of the nitrate of ammonia which interferes with the subsequent reaction, to dissolve it in hot nitric acid, and to continue the treatment according to b.

b. If the calcined oxides dissolve in nitric acid, we evaporate the solution to a syrupy consistency. It has a deep red colour, and contains cerium in the state of a salt of the oxide $\text{Ce}_3\text{O}_4 \cdot 3\text{CeO} = \text{Ce}_6\text{O}_7$. To this semi-fluid mass we add a solution of 5 per cent nitrate of ammonia (thirty to forty times the weight of the oxides), and boil. If no precipitate is formed it is because the solution is too acid; we therefore add a weak solution of ammonia, drop by drop. Each drop will cause the formation of a flocculent violet precipitate, which re-dissolves on agitation up to the moment when a persistent pale yellow precipitate appears. When the supernatant liquor has no longer the least trace of yellow colour, but takes the characteristic violet colour of didymium salts, the reaction is at an end. The precipitate can be filtered and washed with the greatest ease, and when the wash water no longer gives a precipitate with oxalate of ammonium, the precipitate is absolutely free from lanthanum, didymium, and the earths of the yttria group. But the cerium thus obtained only represents a portion, about 75 per cent, of the total contained in the solution. It is not difficult to understand the reason. The action of nitrate of ammonium dissociates the oxide $\text{Ce}_3\text{O}_4 \cdot 3\text{CeO}$, an oxide in which CeO is, by-the-bye, partially replaced by DiO and LaO ; Ce_3O_4 is precipitated as nitrate $(\text{Ce}_3\text{O}_4)_4 \cdot \text{N}_2\text{O}_5$, and CeO remains in solution with the other earths in the state of a neutral salt.

This process—the only one which enables us to obtain cerium completely free from lanthanum and didymium at one operation—can even be made to serve, as we shall show, for a sufficiently accurate method of qualitative separation. The cerium thus obtained, however, still contains an impurity from which it is not easy to separate it. At the same time as the cerium is precipitated, all the thorium in solution comes down also.

Thorium.—This metal, which is only just becoming known, and very little at present, nearly always accompanies cerium in its ores, even in cerite. All the ordinary methods in use will give us thorium free from cerium, but none of them give cerium free from thorium. Such is the case with the two best known of them, the hyposulphite of soda (Chydenius) and the suboxide of copper method proposed by Lecoq de Boisbaudran. We know that the former precipitates barely 85 per cent of thorina, and experiments made on synthetic mixtures have shown us that the latter removes only about 35 per cent. Furthermore, in both cases, a notable quantity of cerium is carried down, but this can be separated by repeating the operation two or three times. It is through not being sufficiently careful to guard against the presence of thorium, and through having accumulated it in successive fractional crystallisations, that we have sometimes found such

* When the oxides contain more than 10 per cent of thorium, it is as well to get rid of the greater part of it, by means of carbonate of ammonia, as will be shown further on.

strange variations in the atomic weight of cerium. The solution of the mixed sulphates of thorium and cerium, containing an excess of the latter, behave on evaporation quite differently to what one would be inclined to expect from the indications found in classic works. It is cerium, the most soluble, which separates out first: this goes to show that an excessively soluble double salt is formed, which crystallises with difficulty; it dries at the ordinary temperature to the condition of a transparent varnish. The analysis of this dehydrated salt leads us approximately to the formula $4\text{SO}_4\text{Ce}, \text{SO}_4\text{Th}$. At the ordinary temperature 100 parts of water dissolve 66 parts of the anhydrous salt.

The best way of getting rid of the thorium, when it is present in any quantity, is to treat the oxalates, or better still the nitrates, with carbonate of ammonium to which has been added a little ammonia. The thorium dissolves with the greatest facility, and after two or three leachings there is only an insignificant quantity (1 per cent) of thorium left.

To remove the last traces we make use of the property we have just mentioned, by crystallising the sulphate, free from free sulphuric acid, at 50° to 60° . The thorina remains in the mother-liquor, and after two or three crystallisations nitride of potassium—the most sensitive known reagent for thorium—will no longer give any precipitate. Cerium thus prepared may be considered as pure, at least within the limits of our actual knowledge, and when transformed into sulphate will serve for the determination of the atomic weight.

(To be continued).

A CRITICAL REVIEW OF THE METHODS OF DETERMINING MINERALS.*

By Dr. JOSEPH W. RICHARDS,
 Of the Department of Metallurgy and Mineralogy of the Lehigh University.

(Concluded from p. 116).

I WILL end this review by describing the methods of determinative mineralogy taught at the Lehigh University, and which my ten years' experience has proven the most certain way of teaching a student to identify minerals.

First of all, the basis is the chemical identification, but with the aid of physical tests brought in at the most profitable stage of the inquiry.

The observer is to first examine the specimen carefully, noting everything that is to be seen or observed by the most simple tests, such as approximate hardness and streak, but not to spend any length of time, more than a minute or two, at such observation. The object of this casual inspection is to determine whether the mineral is similar to any that the observer has seen before; if it is, and therefore suggests one or even several species, the observer is immediately to make a confirmatory test, preferably chemical, and as general as possible, to see if it can possibly be what is suspected. If the test is affirmative, then other confirmatory tests must be applied, with the particular purpose of excluding all related minerals of closely allied composition. If the test is negative, and no other mineral or minerals suggest themselves, then the general method of procedure, based solely on chemical composition, is to be followed.

As an illustration, suppose a mineral appears on casual inspection to be wavelite. The first test is to determine whether it is a phosphate or not. If it is, then alumina, water, and finally fluorine, can be tested for to complete the identification. If not a phosphate, it may have been observed that the mineral glowed strongly and gave a calcium flame, which would suggest *aragonite*, and a test

for carbonic acid would be in order. Or it might have been observed that it fused easily, thus suggesting a radiated zeolite, and tests for silica and water would be in order. If none of these were observed, then the mineral would have to be attacked along broader lines; but if any of these tests have resulted affirmatively, then the observer has profited by his previous knowledge to obtain a short cut to the identification of the specimen. It is evident that the wider the observer's previous acquaintance with numerous varieties of numerous species, the more he will profit by his experience and be able to put it to use in identifying the specimen.

If the observer has no definite idea, from this casual inspection, of what the mineral may be, or if the ideas given by casual inspection were negated by the confirmatory tests, then some regular outline of procedure must be followed, and the following outline is offered as having been used very successfully for several years by your lecturer. Leaving aside the first three items, as being intended for identifying a few simple mineral species, clearing the ground, so to speak, the real classification begins at IV. All the species included in I., II., and III. are included in the later classes.

The outline is as follows:—

Lecturer's Methods.

- I.—If metallic and malleable, look for native metals.
 - II.—If very light and black, examine for hydrocarbons.
 - III.—If it has a taste, examine for chlorides, nitrates, sulphates, &c.
 - IV.—If of metallic, adamantine, or resinous lustre, test in the open tube for S, As, Sb, Se, Te. If present, roast thoroughly, and test by beads, flame, and reduction.
 - V.—If of any other lustre, or if no test is obtained in IV., make bead test for silica. If present, make flame test, &c.
 - VI.—If silica is absent, test for phosphates and borates. If test is obtained, make bead test, reduction, &c.
 - VII.—If P and B are absent, test for carbonates. If test is obtained, make flame test, beads, reduction, &c.
 - VIII.—If CO_2 is absent, test for sulphuric acid. If test is obtained, make flame test, beads, reduction, &c.
 - IX.—If SO_3 is absent, test with KHSO_4 for volatile acids. If test is obtained, make flame test, beads, &c.
 - X.—If volatile acids are absent, make test IV. (open tube), if it has not previously been made.
 - XI.—If open tube has already shown nothing, make the bead tests very carefully, to find the weaker acids, such as Cr, V, U, Ti, Mo. If present, make any other tests.
 - XII.—If weaker acids are absent, substance is probably an oxide. Make any blowpipe tests not already made.
- It will be observed that the outline follows generally the broad lines of the classification of mineral species used by Dana, the primary idea being to class the mineral first by finding its acid constituent. This has the disadvantage of bringing most of the oxides and native metals last in the classification, and makes it desirable that the observer be well informed with those classes by previous acquaintance, as thus much time is frequently saved. If there is any doubt about the lustre, the testing begins at IV.; if the lustre is certainly not of the kinds therein named, it is best to begin at once at V.
- In testing for the acid ingredient, those are tested for first which are most likely to be present (other things being equal), and also those tests are made first which are the quickest to make (other things being equal). Thus, silica is tested for before carbonic acid, because the silicates are more numerous; while phosphates are tested for before carbonates, because the test takes less time.
- After an acid is found, the direction of the testing is towards the basic ingredient. It should be observed, however, that if the tests at any time in any way such information about the mineral that the observer suspects what it is, then let him at once leave the general outline,

* The Journal of the Franklin Institute, cxliv., p. 139.

and by direct tests confirm or deny his suspicion. If, for instance, when testing for phosphorus the barium flame is seen, and the observer then looks at the mineral again and suspects *barite*, then he should test at once for sulphuric acid to affirm or negative his suspicion, without going through the form of testing for carbonic acid. If sulphuric acid is absent, then let him take up the scheme again where he left it, and test in regular order for carbonic acid.

By following these directions, the composition of a mineral, or at least one acid and one base present, can quickly be found. The question then arises, how many minerals can contain the ingredients so far found; and in order to facilitate the search for minerals containing certain elements, I have prepared tables of minerals arranged according to the elements they contain. These tables contain under the heading of each element a classification of all the minerals containing that element, arranged primarily according to the acid ingredients, in the order in which they would be found when following the outline of procedure I have indicated. Under each acid the minerals are further classified according to the other constituents which may be present, arranged in the order in which they are most quickly and surely identified by blowpipe tests, followed by these minerals containing only the acid and the base, or possibly containing some other ingredients not recognisable by blowpipe tests. Wherever, as often happens, a list is obtained of several minerals containing exactly the same ingredients, a small table of physical properties placed to the right gives the means of distinguishing them apart. A sample page is as follows:—

LECTURER'S TABLES (sample page).

Copper.

Physical Properties,
Composition, &c.

Sulphates:—

With Pb:

† Cl: Caracolite.

† Fe: Beudantite.

* : Caledonite, linarite.

With Zn:

† Al: Aluminite (impure).

* : Serpierite.

With Fe:

† Al: Cyanotrichite.

* : Phillipite, pisanite.

With U:

† Ca: Uranocalcite.

* : Voglianite, johannite (H_2O —5 per cent).: Zippelite (H_2O —16 per cent).

With Na:

With K:

With Ca:

With Cl:

With H_2O :

* * :

It will be observed that, granting that the experimenter has found sulphuric acid and copper, if he does not at once identify the specimen by that information, his next step should be to reduce with soda for lead and zinc; if lead is found, then tests for chlorine or iron are in order; and if neither of these be present, the mineral must be caledonite or linarite, which must be told apart by reference to their physical properties. If the reduction with soda has shown nothing, the bead test for iron and uranium is in order. If these are absent, the flame test for sodium, potassium, and calcium is to be made; and if these are absent, the special test for chlorine, and then water. If none of these are present, the mineral will be found in the list following the double asterisks, and distinguished in that list by its physical properties. (The names of these latter minerals and the physical properties are omitted in this sample page for lack of space).

It will be objected to this method that with minerals

which may contain small amounts of many ingredients their classification will be very difficult. This is admitted; but by classifying these minerals under each heading that they can possibly come, as well as under that heading where they must invariably come, the careful observer cannot fail to identify them, no matter what known variety of composition they may show.

The essential idea of this method of working is to use the physical properties as suggestive or confirmatory—suggestive on casual inspection of what the mineral may be, such suggestion to be confirmed by chemical tests; confirmatory after a determination by chemical tests. Conversely, the chemical properties or composition are used as the true determinative factors, to which the physical tests are subsidiary or confirmatory. Several years' practical use in the mineralogy laboratory has demonstrated, as far as my experience goes, the superiority of this method of determination, both in reliability and—taking the average—in quickness.

(At the close of the lecture there was shown a portable specific gravity hydrometer, made by Wood and Comer, of Philadelphia, with especial reference to the use of the mineralogist, and which gives rapidly and accurately the specific gravity of pieces weighing not over 1 gm. Its use in the laboratory work at Lehigh University has demonstrated this claim, and shown that it meets the present need for a cheap, portable, and accurate instrument for determining specific gravities).

THE ATOMIC MASS OF TUNGSTEN.*

By WILLETT LEPLEY HARDIN.

A REVIEW of the literature on the determinations of the atomic mass of tungsten will show that a careful examination of the methods employed is of fundamental importance. Fifteen experimenters have made determinations of this constant, but with widely varying results. The method of investigation, with few exceptions, has been to reduce tungsten trioxide in a current of hydrogen at a white heat, and then to re-oxidise the metal thus obtained. Deviations occur not only in the results of the different experimenters, but also, with few exceptions, in the different observations of the same experimenter. Clarke ("A Re-calculation of the Atomic Weights, 1897") closes his summary of all the work on the atomic mass of tungsten with the following words:—"Further investigation is required in order to fully establish the true atomic weight of tungsten."

Deviations in the results of any atomic mass determinations are due either to an inaccurate method or to experimental errors. It is not always an easy matter, however, to determine the source of error in a given series of variable results; but with several series of results from different experimenters, the question can be decided with a reasonable degree of accuracy. If, for instance, we have several series of results by the same method from different experimenters, and if the different series, with the exception of one, agree with each other, the probability is that the deviations in the one series are due to experimental errors. If, on the other hand, deviations occur in each series, and are more or less similar, the probability is that the method is inaccurate.

Almost every series of results on the atomic mass of tungsten, obtained by the reduction of the trioxide in a current of hydrogen, and by the re-oxidation of the resulting metal, shows a variation between the maximum and minimum results of from one to two units, and in exceptional cases the deviation is much greater. In view of these facts, it seems desirable to make a careful study of the method which has usually been employed in this work, rather than add to the already large number of re-

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xix., No. 8.

sults; for, if the method is unreliable, no experimental skill can make the results trustworthy.

The following summary will show the lack of concordance in the results of the earlier determinations:—

Berzelius (*Pogg. Ann.*, viii., 1, 1826) was the first to determine the atomic mass of tungsten. By reduction of the trioxide he obtained the value 189.6 as a mean of two experiments for the atomic mass of tungsten. The difference between the two results was 3.0. These results and those that follow are calculated on the basis of O = 16.

Schneider (*Fourn. Prakt. Chem.*, l., 152, 1850), working with material which had been carefully purified, obtained two series of results for the atomic mass of tungsten, one by the reduction of tungstic acid and the other by re-oxidation of the metal.

Reduction series.	Oxidation series.
184.18	184.21
183.37	184.16
184.01	183.36
184.28	
184.45	
Maximum diff. .. 0.85	

Maximum diff. .. 1.08

The quantity of material used in these experiments varied from 2 to 6 grms.

Marchand (*Ann. Chem. Pharm.*, lxxvii., 261, 1851) reduced the trioxide of tungsten and re-oxidised the resulting metal; the following results were obtained for the atomic mass of tungsten:—

183.91	Reductions.
183.96	
184.16	Oxidations.
184.51	

Maximum difference.. 0.60

Borch (*Fourn. Prakt. Chem.*, liv., 254, 1851) made seven reductions of tungstic acid in a current of hydrogen, and two oxidations of the metal. The results were as follows:—

Reductions.	Oxidations.
184.10	184.53
182.90	184.32
183.77	
184.10	
183.03	
183.77	
183.91	
Difference .. 0.21	

Maximum diff. .. 1.20

The quantities of material varied from 2 to 8 grms.

By weighing the water obtained in the reduction of tungstic acid in hydrogen, Riche (*Fourn. Prakt. Chem.*, lxi., 10, 1857) obtained the value 174 for the atomic mass of tungsten as a mean of two experiments. The difference between the two was 1.78.

Dumas (*Ann. Chem. Pharm.*, cxiii., 23, 1860) reduced the trioxide of tungsten in hydrogen and obtained the following results for the atomic mass of tungsten:—

184.00
183.42
184.16
183.76
183.62
184.80
184.16
184.08

Maximum difference = 1.38

The quantities of material varied from 1 to 4½ grms. Bernoulli (*Pogg. Ann.*, cxi., 573, 1860) reduced tungstic acid at a very high temperature in a current of hydrogen. The results were as follows:—

Reduction series.	Oxidation series.
186.78	186.81
185.86	187.94
186.75	186.77
186.81	186.76
186.70	
177.73	Maximum diff. = 1.18

Maximum diff. = 9.08

The maximum difference in the two series is 10.21. He found that the greenish coloured oxide gave the same results as did the yellow oxide.

Persoz (*Ann. Chim. Phys.*, [4], i., 93, 1864) made two reductions of the trioxide of tungsten and obtained concordant results.

183.93
183.94

Difference = 0.01

Scheibler (*Fourn. Prakt. Chem.*, lxxxiii., 324, 1861), from determinations of the water in barium metatungstate, obtained the value 184.00 for the atomic mass of tungsten. Maximum difference = 1.03.

Zettnow (*Pogg. Ann.*, cxxx., 30, 1867) obtained the value 184.08 for the atomic mass of tungsten as a mean from four analyses of the tungstate of iron. From silver tungstate he obtained the value 183.80.

Roscoe (*Ann. Chem. Pharm.*, clxii., 368, 1872) made three reductions and two oxidations of the same sample of material, beginning with 7.8840 grms. of tungstic acid. The results were as follows:—

Reductions.	Oxidations.
182.72	182.49
183.71	183.87
183.97	

Difference = 1.38

Maximum diff. = 1.25

From two analyses of tungsten hexachloride, Roscoe obtained the value 184.25 for the atomic mass of tungsten.

Waddell (*Am. Chem. Journ.*, viii., 280, 1886), from carefully purified tungstic acid, obtained by reduction in hydrogen the following values for the atomic mass of tungsten:—

184.55
184.37
184.59
184.00
183.67

Maximum difference = 0.92

The quantities of material varied from 1 to 4½ grms.

The material used in the work of Pennington and Smith (*Zeit. Anorg. Chem.*, viii., 198) differed from that of all the preceding experimenters, in that the last traces of molybdenum were removed by gently heating the tungstic acid in a current of hydrochloric acid gas. The method of operation was also somewhat different from those of the earlier experimenters. The metallic tungsten used in the oxidations was obtained by the reduction of tungstic acid in a platinum crucible at a white heat, in a current of hydrogen, which was conducted through the lid of the crucible. The mean of nine results from the oxidation of the metal is 184.921 for the atomic mass of tungsten. The maximum difference in the series is 0.043. The quantities of material used varied from 0.43 to 1.08 gm.

Smith and Desi (*Zeit. Anorg. Chem.*, viii., 205) weighed the water obtained in the reduction of tungstic acid, and from that calculated the atomic mass of tungsten. The mean of six determinations is 184.704. Maximum difference 0.071.

Schneider (*Fourn. Prakt. Chem.*, liii., 288, 1896) made a second series of reductions and oxidations. The material used in these experiments was freed from molybdenum by gently heating the tungstic acid in a cur-

rent of hydrochloric acid gas. The values obtained for the atomic mass of tungsten were as follows:—

Reduction series.	Oxidation series.
184·14	184·00
183·98	183·92
183·96	184·04

Maximum diff. = 0·18 Maximum diff. = 0·12

The quantities of material varied from 2 to 6 grms. Shinn (Thesis, University of Pennsylvania, 1896) obtained by oxidation of metallic tungsten the following values for the atomic mass of tungsten:—

184·72
184·96
184·75
185·22

Maximum difference = 0·48

The quantities of material used varied from 0·10 to 0·22 grm. of metal.

A glance at the foregoing results will show a remarkable variation. The extremely high value obtained by Berzelius is supposed to be due to the presence of alkaline impurities in the material used.

The observations of Schneider, Marchand, Borch, Dumas, and Waddell are very similar. The method of operation was the same in each case, and the material used was purified with considerable care. It is difficult to account for the variations which occur throughout these results. Molybdic acid is probably the only impurity that could have contaminated the material used in the experiments. Such an impurity would probably have lowered the separate results by the same amount, and hence would not have produced the variations. The deviations between the maximum and minimum results of the different experimenters are as follows:—Schneider 1·08, Marchand 0·60, Borch 1·63, Dumas 1·38, and Waddell 0·92.

The results of Riche and Bernoulli differ widely from those obtained by other experimenters. The extremely low value obtained by the former is probably due to the method. The high results obtained by Bernoulli are more difficult to explain. The material used was carefully purified. The tungstic acid used in some of the experiments was of a greenish tinge. Some have assumed that this material was incompletely oxidised, and in this way account for the high results. Bernoulli found, however, that the greenish coloured oxide and the yellow oxide gave the same results when reduced. Furthermore, neither the colour of the original oxide nor the state of oxidation could affect the results obtained in the re-oxidations of the metal. The results obtained by the latter method are higher than those obtained in the reductions. In view of these facts, the explanation which has been offered to account for these high results is entirely unsatisfactory.

Scheibler's results on barium metatungstate show a variation of more than one unit, and it must be added that the results obtained by the determination of the barium and tungsten in this salt were still more variable and were not used by Scheibler in calculating the atomic mass of tungsten. The two short series of results on ferrous and silver tungstates by Zettnow are reasonably concordant.

Roscoe's experiments on the same sample of material are rather interesting. The material was reduced and re-oxidised several times without being removed from the porcelain boat. The maximum difference in a series of five results is $1\frac{1}{2}$ units. If the method employed by Roscoe is accurate, it is difficult to account for this variation.

The most concordant series of results on the atomic mass of tungsten is that of Pennington and Smith. The value obtained is higher than that obtained by most ex-

perimenters. Schneider (*Journ. Prakt. Chem.*, liiii., 283, 1896) has attempted to account for the high values obtained in these experiments; but, inasmuch as these results agree very closely with those obtained by Smith and Desi and Shinn, it is useless to offer an explanation for this high value until the true atomic mass of tungsten is known with greater certainty, at least until a series of concordant results has been obtained which differs from these.

Schneider's last determinations consist of two series of results, each series containing three observations. From these two short series of reasonably concordant results, Schneider concludes that the atomic mass of tungsten may be safely considered equal to 184·00. The evidence, however, is far from satisfactory. In view of the wide variations in the earlier determinations, the number of results in these experiments is entirely too small to establish anything with certainty with regard to the true atomic mass of tungsten. This fact is shown in the work of Waddell, who made five determinations. The maximum variation in the first three observations was only 0·22, while in the series of five the variation was 0·92. The same is noticed in the work of other experimenters. And in the present investigation, consisting of more than sixty determinations, a series of five concordant results were sometimes obtained, after which considerable variation was obtained. Attention will be called to this fact again in the discussion of the following observations.

(To be continued).

EXPERT TESTIMONY.*

By WILLIAM P. MASON.

It will be remembered that a would-be facetious barrister once remarked that prevaricators might be properly arranged in an ascending series, to wit, ordinary fibbers, liars, and experts,—an arrangement which I fear meets with the approval of many members of the bench and bar to-day. The cause for such harsh classification is not so very far to seek. It is based upon ignorance on the part of the bar, and at times upon what is worse than ignorance on the side of the "expert." With the culpable acts of the pseudo scientist we cannot waste our time. That he merits prompt condemnation is axiomatic; but a word is wanted touching upon what may be termed the ignorance of the court.

"When I take my place upon the witness stand," said a prominent toxicologist once to me, "I can never predict in what shape I shall be upon leaving it," a feeling with which most of us can, I fancy, sympathise pretty keenly.

Is it that we fear exposure of the weak points in our professional armour? Do we dread to say in public "I do not know?" Hardly that, I take it. We are now possessed of so very little of that which one day may be known, that no true scientist hesitates for an instant to plead legitimate ignorance. What really troubles us upon cross-examination is that the court does not speak our language, a language often quite difficult of direct translation; that it is but rarely schooled in the principles of our science; and that, in consequence, it frequently insists upon categorical answers to the most impossible kind of questions.

The hypothetical questions showered upon the expert witness are sometimes veritable curiosities, so peculiar are they in their monstrosity. Who among us but has felt that the layman, who has simply to testify to observed facts, has an easy time of it indeed, when compared with him from whom there is expected an opinion under oath?

* An Address by Vice-President William P. Mason, Chairman of Section C, before the Section of Chemistry, American Association for the Advancement of Science, Detroit Meeting, August, 1897.

All scientific men are willing and anxious to have their work scrutinised carefully by their peers; but to be exposed to the one-sided criticism frequently encountered at the bar is quite another matter; for it must be remembered that after the adverse counsel has opened up what appears to be a glaring inconsistency in the testimony, the re-direct examination may utterly fail to repair the breach, because of a lack of familiarity with a technical subject on the part of the friendly attorney.

This leaves the witness in the unenviable position of disagreeing with the general drift of his own testimony, while it deprives him of suitable means of insisting upon its revision and correction.

According to the writer's view there is but one way to escape such dilemma, and that is by direct and immediate appeal to the judge, urging that the oath taken called for a statement of the whole truth, and not the misleading portion already elicited.

To illustrate how serious a matter the partial testimony of an expert witness may be, and to show also to what extent lawyers may go who look only to the winning of their causes, permit me to refer to an already reported poison case in which I was employed by the people. It may be roughly outlined as follows:—

Much arsenic and a very little zinc were found in the stomach.

The body had not been embalmed, but cloths wrung out in an embalming fluid containing zinc and arsenic had been spread upon the face and chest.

Medical testimony showed that no fluid could have run down the throat. Knowing the relative proportions of zinc and arsenic in the embalming fluid, the quantity of arsenic found in the stomach was twelve times larger than it should have been to have balanced the zinc also there present, assuming them to have both come from the introduction of the said embalming fluid by cadaveric imbibition. Other circumstantial evidence was greatly against the prisoner.

At the time of my appearing for the people, on the occasion of the first trial of the case, my direct testimony brought out very strongly the fact that a fatal quantity of arsenic had been found in the stomach, but no opportunity was given me to testify to the presence of the zinc found there as well, although the fact of its existence in the body was known to the prosecution through my preliminary report. Through ignorance of the nature of such report on the part of the defence, no change was made in the character of my testimony during the cross-examination, and I was permitted to leave the witness-stand with a portion of my story untold. No witnesses were called for the defence, and the case was given to the jury with the darkest of prospects for the prisoner.

For many reasons, unnecessary to recount here, I was distinctly of the opinion that murder had been committed, but I felt nevertheless that common justice demanded that the prisoner should have been entitled to whatever doubt could have been thrown upon the minds of the jury, no matter how far-fetched the foundations for such doubt might have been.

The first trial having resulted in a disagreement of the jury, I was pleased to learn, before the second hearing of the case began, that the defence was prepared to go into the question of the embalming fluid; for the responsibility of permitting only a part of what I knew to be drawn from me, to the entire exclusion of the remaining portion, was greater than I wished to assume. The nature of my report to the coroner having been established, and certain opinions relating thereto having been fully ventilated, the jury were possessed of "reasonable doubt" and acquitted the prisoner. What now were the duties of the expert upon the occasion of the first trial of this case, and how should he have construed the meaning of his oath?

One eminent legal light, to whom the question was referred, held that the expert was distinctly the property of the side employing him, and that his duty was simply to answer truthfully the questions put to him, without at-

tempting to enlighten the court upon facts known to him, but not brought out by the examination, no matter how vital such facts might be.

Another held that although the above course would be proper in a civil case, yet, in a matter involving life and death, the witness should insist upon the Court becoming acquainted with his whole story. Do not such differences in legal opinion make it very desirable that the expert, at least in capital cases, should be the *employé* of the bench rather than of the bar, in order that whatever scientific investigations are made may be entirely open to public knowledge and criticism?

Although the expert should earnestly strive to have what he has to say presented in the best form, he must remember that to secure clearness, particularly before a jury, technicalities should be reduced to a minimum. To a degree they are unavoidable, but let them be as few as possible. Illustrations should be homely and apt, capable of easy grasp by the jury's minds, and, if possible, taken from scenes familiar to the jury in their daily lives.

It is an unfortunate fact that the expert must be prepared to encounter in the Court-room not only unfamiliarity with his specialty, but also deep-rooted prejudices and popular notions hoary with age and not to be lightly removed from the mind by the words of a single witness. As President Jordan has well said, "There is no nonsense so unscientific that men called educated will not accept it as science," and, let me add, they will calmly attempt to shove the burden of proof upon the scientific man who is opposed to their views. Sanitary experts, in particular, run up against all sorts of popular superstitions, and are inveighed against as "professors" by those who consider themselves the "practical" workers of the time; and, let it be noted, the burden of proof is uniformly laid upon these "professors'" shoulders, while the most astounding and occult statements made by the "practical" men may be received without verification.

One source of trouble, which perhaps is peculiar to the water expert, lies in the impossibility of utilising analytical results such as were made many years ago.

Those who are not chemists fail to grasp the fact that the examination of water may not be looked upon from the same point of view as the analysis of an iron ore. The statement that water analysis is but of recent birth, and that it is yet in its infancy, is hard for them to appreciate, holding, as they naturally do, that what was true twenty years ago must be true to-day, if science does not lie.

A pit into which many an expert witness falls is prepared for him by insidious questions leading him to venture an opinion upon matters outside of his specialty. It is a fatal error to attempt to know too much. Terse, clear answers, well within the narrow path leading to the point in question, are the only safe ones; and when the line of inquiry crosses into regions where the witness feels himself not truly an expert, his proper course is to refuse to testify outside of the boundaries of his legitimate province.

Unfortunately the expert is as often invited to take these collateral flights by the side employing him as by the opposition. Affidavits in submitted cases are commonly written by the lawyers and not by the expert, although they are, of course, based upon his reports. In the strength of his desire to win the case, the lawyer often prepares a much stronger affidavit than his witness is willing to swear to.

The writer has had no little difficulty just at this point, and has had plenty of occasion to observe the irritation displayed by counsel upon a refusal to endorse statements which have been "too much expanded."

Every expert-witness, especially in his early cases, is sure to have adverse authorities quoted against him; therefore it behoves him to be so familiar with the literature of his subject as to be capable of pointing out that such and such a writer is not up to date, or that such and such a passage, if quoted in full, would not bear the ad-

verse construction that its partial presentation carries. When the expert reaches a position of such prominence that he can state a thing to be so because he says it, irrespective of whatever may be written on the subject to the contrary, his course then is greatly simplified; but long before he attains that altitude he will have put himself upon record in many cases, and happy for him if the record so made be such as cannot be quoted to his disadvantage.

"If I had only not written my first book," is the reflection of many a distinguished author; while one of the great masters of music, referring to an opera, said "It is one of my early crimes."

Above all things, the expert "should provide things honest in the sight of all men."

It is well for him to be deeply interested in his case, to feel in a measure as if it were his own; but it is unwise in him to become so partisan as to let his feelings affect his good judgment, and it would be indeed criminal should he permit his interest in any way to contort the facts.

Before the case is brought to a final hearing it may be apparent that experiments before the Court are possible, and they may be demanded by the counsel in charge of the case. If such experiments be striking, easy of execution, and not too long, by all means make them.

Practical illustrations, particularly such as involve some fundamental principle, have great weight with the Court; but these illustrations must not be such as would turn the Court-room into a temporary laboratory and involve the loss of much time in vexatious waitings.

Such experiments as are determined upon should be thoroughly rehearsed beforehand, no matter how simple they may be; for, of all failures, the Court-room experiment which declines to "go off" is perhaps the most dismal.

This brings to mind a kindred topic upon which there should be a word of caution: laboratory experiments which work to perfection may utterly fail when expanded to commercial proportions, so that it is wise to bear in mind the danger of swearing too positively as to what will happen in large plants, when the opinion is based only upon what is observed to occur upon the smaller scale. Like conditions will, of course, produce like results; but it is marvellous how insidiously unlooked-for conditions will at times creep into one's calculations, and how hard it is even to recognise their presence.

When preparing his case for presentation, the expert often errs in not dwelling more largely upon certain points because he thinks them already old and well known. To him they may be old, but to the public they may be of the newest. Not only is the public unequally posted with the specialist, but what it once knew upon the subject may have been forgotten. It is well therefore to insert, in a special report, matters that would be properly omitted from a paper prepared for a professional audience.

Sanitary problems are of especial interest to the public, but the amount of ignorance, or rather false knowledge, displayed concerning them is surprising and often difficult to combat. The sanitarian is not unfrequently called upon suddenly to defend a position involving complex statistics; and because the data cannot be forthwith produced, the inference is drawn that his points are really without facts to support them, and that they are consequently not well taken.

Long before he gets into court, particularly if the time for preparation of the case be short, the expert may well "pray to be delivered from his friends." He may receive a peremptory order by telegraph to "determine the mineral qualities of this rock," when the telegram should have read "Assay this ore for silver," and later it may be a matter of surprise that a quantitative knowledge of the copper present was not obtained while passing along the line for the determination of the silver; for it is generally not known that the complete analysis of anything is quite rare and correspondingly tedious and expensive.

Toxicologists who hear me may call to mind some case

involving a search for the presence of an alkaloid, strychnia for example, during which search the district attorney, in his eagerness for information, may have asked to know what the indications were as to the presence of the poison, at a time when the extraneous organic matter was not nearly removed. He may have wished no final report, but only the simple probabilities, whereon to base a possible arrest. Such requests are very common, and are akin to a demand for a proof of the pudding during the early baking, when we all know that such proof comes at a much later stage of the proceedings.

Finally, "When doctors disagree who shall decide?"

This question is often very vigorously settled by the jury, as was instanced in a recent celebrated murder trial in New York city. In that case what the experts had to say on either side was simply thrown overboard as a whole, and the finding was based upon the testimony of the remaining witnesses.

What can be said upon this question of the disagreement of expert witnesses? First, it must be noted, they are far from being the only class of people who fail to agree, and that too on very important subjects. Do my hearers think it would be a very difficult task to find a small army of men who would testify very variously and very positively upon questions of politics or religion? Would it be hard to find "good men and true" who would give under oath greatly differing opinions concerning the propriety of instituting free trade or establishing an inheritance tax? Experts are subject to the same errors of judgment as befall the rest of professional humanity, and, when their opinions clash, they are entitled to the same respect that we grant to the members of the Bench when they hand down the decision of a divided court.

One fruitful opportunity for disagreement always arises when questions are brought into court touching upon matters newly discovered, and apart from the well-beaten path of common professional knowledge. Doubt is often left upon the minds of those seeking the light, even when the testimony is given by the specialist who originally developed the new point in question, for one cannot be expected to be thoroughly educated in that which he has himself but recently discovered.

Many of us have dreaded to see the "ptomaines," or putrefactive alkaloids, make their way into court with their mystifying influences upon judge and jury and their tendency to protect crime. Now that they are in, what is to be the end? Even with no "ptomaine theory" possible, the ptomaine form of argument is not unknown. The writer was once asked, in an arsenic case, whether he was willing to swear that at some future time an element would not be discovered giving the stated reactions now called arsenical? Such nonsense is of course instituted to impress the jury, and is suggested by similar questioning in the alkaloid cases.

A recent and somewhat amusing instance arose from an attempt to introduce the rather new conception of "degeneracy" into a murder trial. The defence sought to show that the prisoner was a "degenerate," and offered expert testimony as to the meaning of the term and as to the signs whereby such a condition was to be recognised; whereupon the prosecution called attention to the fact that the defendant's experts themselves exhibited every one of the signs in question.

Having said all that he was to say, and having stated it to the best advantage, should the expert depend upon the stenographer's so recording it as to allow of its being used in future without correction? Decidedly not.

The average stenographer is unfamiliar with technical terms, especially such as are chemical, and the witness who fails to supervise the minutes may find out later that he was sworn to a most remarkable array of "facts." The writer once discovered that he had recommended, as a very efficient method of purifying a city water, the filtering of the entire supply "through a layer of black mud." Not to take your time further, let us summarise what has thus been briefly said:—

The expert witness should be absolutely truthful, of course; that is assumed, but beyond that he should be clear and terse in his statements, homely and apt in his illustrations, incapable of being led beyond the field in which he is truly an expert, and as fearless of legitimate ignorance as he is fearful of illegitimate knowledge.

Mounting the witness-stand with these principles as his guide, he may be assured of stepping down again at the close of his testimony with credit to himself and to the profession he has chosen.

NOTICES OF BOOKS.

Organic Chemical Manipulation. By J. T. HEWITT, M.A., D.Sc., Ph.D., F.C.S. (London and Berlin), Professor of Chemistry in the East London Technical College. With 63 Illustrations. London and New York: Whittaker and Co. 1897. Pp. 260.

It is very satisfactory to find the evidently increased attention now paid to organic chemistry, both in its analytical and its synthetical phases. Dr. Hewitt, in the work before us, is contributing ably to meet the demand thus springing up.

The first chapter discusses the purification of organic substances. In the second we come to the ultimate analysis of organic compounds, the determination of equivalent and molecular weights; whilst the fourth chapter treats of the estimation of special groups of atoms in organic compounds.

The second part enters upon the preparation of organic substances, classified under the heads of "Compounds of the Fatty Series" and "The Aromatic Hydrocarbons and their Derivatives."

After a careful inspection, we feel fully warranted in recommending this little work to the earnest student.

Manueli Hoepli. Metallic Alloys and Amalgams; Aluminium, Nickel; the Precious Metals and their Imitations; Bronze, Brass, Coins, Medals, Solders. By the Engineer S. GHERSI. Milan: Ulrico Hoepli.

We find here a very complete account of alloys in general, their composition and properties. Special attention has been paid to the influence—generally deleterious—of traces of foreign metals. The tin of Banca approaches very closely to chemical purity, that of Queensland taking the second place. The coppers of Australia and of Lake Superior differ little in their degrees of purity.

The following alloy exhibits a curious modification of colour; it is purple with ruby-red reflections. The compound of 22 per cent of gold with 78 per cent of aluminium—known as Margot's alloy—is perhaps the most beautiful, but it is deficient in malleability. Margot is of opinion that its colour is due to microscopic crystals of alumina disseminated in the mass; but this is open to doubt. The yellow alloy of platinum and aluminium can, by a modification of the proportions, be rendered violet or greenish.

The experiments of Krupp are in favour of the use of iron-nickel for artillery and metal plating. If steel is of an inferior quality it is not improved by the addition of nickel.

A rose-coloured alloy is obtained with 750 parts gold, 200 parts silver, and 50 parts of copper.

Tables for Gas Analyses, Gas Volumetric Analyses, Determination of Nitrogen, &c. ("Tabellen für Gas Analysen, Gas Volumetrische Analysen, Stick Stoffbestimmungen, &c."). By Prof. Dr. G. LUNGE. London, Edinburgh, and Oxford: Williams and Norgate. 1897.

A SET of useful analytical tables, conveniently arranged for laboratory use.

CORRESPONDENCE.

OCCURRENCE OF VANADIUM.

To the Editor of the Chemical News.

SIR,—*Apropos* of the description of the occurrence of vanadium in rutilite by Hasselberg (see CHEMICAL NEWS, vol. lxxvi., p. 112), the following analysis of a sample of rock from South Africa may be of interest. It was sent here for analysis as a sample of iron ore:—

SiO ₂		0.99
Al ₂ O ₃		2.06
Fe ₂ O ₃		72.85
FeO		5.24
MnO		0.20
CaO		0.40
MgO		0.54
TiO ₂		15.00
V ₂ O ₅		0.59
NiO		0.30
Cr ₂ O ₃		0.26
Combined H ₂ O		1.37
Moisture		0.58
Phosphorus		nil
Sulphur		trace
Arsenic		nil

100.38

—I am, &c.,

F. W. DAW, A.R.C.Sc., F.C.S.

Ebbw Vale, September 4, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxv., No. 7, August 16, 1897.

Dr. Ferrand made, through the mediation of Armand Gautier, a claim of priority as regards vaccination against cholera.

Researches on the Simple Cathodic Rays.—H. Deslandres.

Action of Röntgen Tubes behind Screens Opaque to X Rays.—Abel Buguet.—The radiations concerned do not come essentially from the solid bodies employed in the experiments. They are attenuated when the sensitive surface is more or less masked by sheets of paper, aluminium, tin, silver, or lead. They did not traverse a sheet of lead of 0.5 m.m. The radiations may be used in the radiography of partially opaque bodies in cases where the direct action of the X rays is arrested by impassable obstacles. The phenomenon is not affected by a magnetic field, nor by a powerful current of air playing over the surface of the sensitive body.

Bulletin des Travaux de la Société de Pharmacie de Bordeaux. July, 1897.

[Analysis of the Oil of American Black Walnuts (*Juglous nigra*, L.).—MM. Barthe and Boutineau.—The tree producing these nuts was imported into Europe in 1656. It is now common in the Gironde and Lower Pyrenees, where it grows to a considerable size. And although the nut itself is not so agreeable to the taste as the ordinary walnut, on account of its slight flavour of turpentine, it gives a useful and quickly-drying oil. One

kilogram. of the kernel gives, as a rule, 470 grms. of oil; its specific gravity is 0.929 at 15° C.; its acid index, expressed in m.grms. of caustic potash, is 4.12; its index of saponification is 195; its index of refraction is +26° at 15° C. in Jean's refractometer; and it dissolves in absolute alcohol to the extent of 6 grms. per litre.

Examination of the Official Solution of Perchloride of Iron.—E. Falières.—This solution should contain 26 per cent of anhydrous ferric chloride, or 8.96 per cent of iron; its density is then 1.26. It may, however, be adulterated and its density then brought up to the right point by the addition of salts of soda or potash. The author has sought for a rapid method of estimating its purity, and found that this could be best done by alkalimetric titration of the quantity of acid in combination with the iron, and the estimation of the chlorine by nitrate of silver.

Possible Manner of the Formation of the Fossil Phosphates of Lime in the Province of Oran.—M. Dion.—The author concludes that these fossil phosphates have an aqueous origin; that they are formed, from above, downwards, by the action of infiltrating rain-water; and that animal remains were the only source of phosphorus in these deposits, as no phosphates belonging to the crystalline minerals are to be found in the neighbourhood.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xvii.-xviii., No. 13.

M. Gautier presented, on behalf of M. Hélier and himself, the researches they have carried out, on the combination of chlorine and hydrogen under the influence of light and heat.

M. Villiers pointed out a method of oxidation of minerals, and of organic compounds of the fatty series; and of substitution for the compounds of the aromatic series, founded on the property of salts of manganese, of developing or facilitating oxidation when introduced into an oxidising medium. The oxidation of oxalic acid, by hydrochloric and nitric acids, under the influence of sulphate of manganese, was exhibited.

M. Hanriot presented, on behalf of himself and M. Reynaud, the continuation of their researches on isoxazolones.

M. Le Chatelier found in the residue left, after the action of water on carbide of calcium, besides carbide of silicon, silicide of iron and silicide of calcium.

M. Béal presented a note on behalf of M. Prud'homme, who, in studying the action of a mixture of soda and dilute alcohol on the *p*-nitrodiamidotriphenylmethanes, found that a migration of one of the atoms of oxygen of the NO₂ group was produced, and that there was formed carbinol, or the base of a diamidotriphenylmethane, having the group NO in a position similar to that in the benzenic nucleus, not aminised. His researches also confirm the formula attributed to the fuchsines by M. Rosenstiehl.

Liquefaction of Fluorine.—H. Moissan and J. Dewar.—Already inserted in full.

Notes on the Application of Acetylene to Lighting Purposes.—L. M. Bullier.—These experiments have been carried out on pure acetylene, and on mixtures of acetylene with air, nitrogen, carbonic acid, carbonic oxide, hydrogen, and coal-gas, and they prove that if we cannot generally replace coal-gas by acetylene, in all its applications to lighting purposes, the addition of foreign gases will render its use much simpler, and enable us to obtain results which can now only be obtained with great difficulty, and this without the aid of recuperative apparatus or anything delicate and costly.

On some New Methods of Transforming Paranitrodiamidotriphenylmethanes into Fuchsines, or into Bases of corresponding Fuchsines.—M. Prud'

homme.—The author has tried four methods, viz.:—In an acid medium, with heat; in an acid medium, in the cold; in an alkaline medium; and with sulphide of sodium. He finds the latter method the most advantageous, both as regards quality and quantity; but it does not appear to be possible to obtain more than 60 per cent of the theoretical quantity available: this is attributed to a partial reduction of the base into leucobase. He has demonstrated the existence of two forms of the bases of fuchsines, which is not in contradiction to the observations of M. Rosenstiehl or of H. Weil.

MISCELLANEOUS.

British Association for the Advancement of Science.—The following are the names of the Officers and Committee of Section B (Chemical Science) at the Toronto Meeting of the British Association:—

President—Prof. W. Ramsay, F.R.S.

Vice-Presidents—Prof. G. F. Barker; Prof. F. W. Clarke; Prof. H. B. Dixon, F.R.S.; W. R. Dunstan, F.R.S.; Prof. B. J. Harrington; Prof. E. W. Morley; Prof. W. H. Pike; Prof. I. Remsen; Prof. W. C. Roberts-Austen, F.R.S.

Secretaries—Prof. W. H. Ellis; Arthur Harden (Recorder); Charles A. Kohn; Prof. R. F. Ruttan.

Committee—Prof. W. W. Andrews; Prof. H. E. Armstrong, F.R.S.; Prof. B. Brauner; Prof. C. Le Neve Foster, F.R.S.; Prof. W. L. Goodwin; A. Vernon Harcourt, F.R.S.; O. S. James; F. B. Kenrick; Prof. Loeb; Prof. H. McLeod, F.R.S.; Prof. Meldola, F.R.S.; Prof. Meslans; W. L. Miller; Prof. Nef; H. Ramage; W. W. Randall; Prof. T. W. Richards; F. J. Smale; Prof. A. Smith; A. Stansfield; Prof. W. C. Williams.

The Papers brought before the Section were as follows:—

President's Address—"An Undiscovered Gas."

Prof. W. W. Andrews—Reform in the Teaching of Chemistry.

Report of the Committee on the Teaching of Science in Elementary Schools.

Report of the Committee on Preparing a New Series of Wave-length Tables of the Spectra of the Elements.

Report of the Committee to inquire into the Proximate Chemical Constituents of the various kinds of Coal.

Report of the Committee on the Isomeric Naphthalene Derivatives.

Report of the Committee on the Action of Light on Dyed Colours.

Prof. Ramsay—Helium.

Prof. Brauner—Contributions to the Chemistry of the Rare Earth Metals.

Prof. Brauner—The Atomic Weight of Thorium.

Prof. T. W. Richards—The Atomic Weights of Cobalt and Nickel.

M. Travers—The Occurrence of Hydrogen in Minerals.

Prof. W. N. Hartley and H. Ramage—Spectroscopic Examination of Minerals and Metals.

Prof. Meslans—Demonstration of the Preparation and Properties of Fluorine.

Profs. Moissan and Dewar—The Properties of Liquid Fluorine.

Prof. Ramsay—Demonstration of the Spectra of Helium and Argon.

Dr. J. Waddell—The Permeability of Elements of Low Atomic Weight to the Röntgen Rays.

Dr. J. H. Gladstone and W. Hibbert—Continuation of Experiments on Chemical Constitution and the Absorption of X Rays.

Dr. W. J. Russell—On the Action exerted by Certain Metals on a Photographic Plate.

Prof. H. B. Dixon—Photographs of Explosion Flames.
F. P. Dunnington—Titanic oxide.
F. P. Dunnington—Deliquescence and Efflorescence of Certain Salts.
J. Waddell—Notes on Concentrated Solutions of Lithium and other Salts.
W. L. T. Addison—The Formation of Crystals.
E. C. C. Baly—A Compound of Ozone and Mercury.
J. W. Walker—The Interaction of Hydrobromic and Bromic Acids.
F. T. Shutt—The Composition of Canadian Virgin Soils.
Prof. W. H. Ellis—Analysis of some Pre-carboniferous Coals.
Prof. P. C. Freer—The Constitution of Aliphatic Ketones.
Prof. J. U. Nef—The Chemistry of Methylene.
Dr. A. Lehmann—Formation of a Benzene Ring by Reduction.
Dr. C. A. Kohn—Condensation Products of Aldehyds and Amides.
Dr. Hugh Marshall—A New Form of Bunsen-burner.
Prof. W. C. Roberts-Austen—Molecular Movement in Metals.
Dr. T. K. Rose—The Causes of Loss Incurred in Roasting Gold Ores containing Tellurium.
H. C. Jenkins—The Behaviour of Lead and of some Lead Compounds towards Sulphur Dioxide.
Dr. W. L. Miller and T. R. Rosebrough—The Vapour-Tensions of Liquid Mixtures.
Dr. C. A. Kohn—The Electrolytic Determination of Copper and Iron in Oysters.
Prof. Henri—The Nitro-Alcohols.
Prof. W. W. Andrews—The Plaster of Paris Method in Blowpipe Analysis.
R. Ransford—Some Experiments with Chlorine.
Report of Committees—
The Electrolytic Methods of Quantitative Analysis.
Isomeric Naphthalene Derivatives.
The Direct Formation of Haloids from Pure Materials.
The Bibliography of Spectroscopy.
The Carbohydrates of Barley Straw.

Schools of Chemistry, &c.—The following information was received too late for insertion in the "Students' Number":—

SHEFFIELD TECHNICAL SCHOOL (UNIVERSITY COLLEGE, TECHNICAL DEPARTMENT).—Lecturer in Chemistry, F. Ibbotson, B.Sc., F.C.S.; Professor of Metallurgy, J. O. Arnold; Lecturer, A. McWilliam, A.R.S.M.; Demonstrator and Lecturer on Fuel, F. K. Knowles. The work is divided into two departments:—I. The Technical Department of the University College, including Mechanical, Electrical, Civil, and Mining Engineering; Metallurgy. II. The Evening Department, for providing Technical Instruction for persons engaged during the day in the local industries. The courses of Evening instruction include Magnetism and Electricity, Inorganic Chemistry, Experimental Laboratory, and Electrical Engineering. The Metallurgical Department has been equipped with a view to thoroughly meeting the requirements of the local industries. The Laboratory is fitted with the most modern apparatus for metallurgical analysis, more especially with appliances for the rapid and accurate chemical examination of Iron and Steel, Fuel, and Refractory materials. It also contains a complete pyrometric installation, and a new laboratory for the study of the micrographic analysis of metals has been completed and fully equipped with specially designed microscopes, by Ross, polishing tables, driven by an electric motor, etching appliances, incandescent light for evening work, &c. The School is now the most complete of its kind for teaching the practical manufacture, the chemical constitution, and the physical properties of steel. Special attention is given to the determination of the microscopic

constituents of steel. Although the chief industry of the district occupies the central position in the course of instruction, general metallurgy is not neglected, but is dealt with in a separate syllabus, dealing with metals (other than iron and steel) used in the arts. Students are thus enabled to select and at once enter upon a course of scientific metallurgical training of immediate practical utility. They may take up and work through any portions of the course, but certificates will be granted only to those who follow the prescribed courses and pass the necessary examinations. The course of study is as follows:—Preparatory Chemistry, Metallurgy Lectures, Fuel Lectures, Practical Metallurgy, Practical Fuel Course, Geology and Mineralogy, and Electro-Metallurgy.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1974.

ON THE PROPERTIES OF NITROBENZENE.

By RICHARD J. FRISWELL.

DURING the severe winter of January, 1886, a drum of nitrobenzene crystallised, and I succeeded in removing from it a large, tabular, transparent crystal, 2½ inches long, 1 inch broad, and ¾ inch thick, weighing over 15 grms. It was irregular, had no faces, and resembled a mass of ice in appearance. I took advantage of this circumstance to make determinations of the specific gravity of solid nitrobenzene. An Oertling balance was placed in the open air, and allowed to remain until the temperature of an accurate thermometer registered 3°5', and the crystal was then weighed in air and in water in the usual manner.

Weight in air, 15·849 grms.

Weight in water, 4·059 grms.

Weight of water displaced, 11·790.

Temperature of water, 15°.

This gives the sp. gr. as 1·3439, which is correct to ±0·0001.

A large portion of the crystalline mass was then freed from liquid and fused in a paper filter; during the fusion a small quantity of moisture from the air condensed on the melting crystals, but these traces of water were intercepted by the filter, which was rendered waterproof by being saturated with the melted nitrobenzene.

The sp. gr. of the liquid nitrobenzene was ascertained by a very accurate Westphal balance. At 3·8° this was found to be 1·2210, compared with water at 15°, for which temperature the balance is standardised.

During the cooling to 3·8° the liquid became slightly turbid, indicating that the moisture condensed on the fusing crystals had been dissolved to a minute extent.

Correcting the above numbers for water at 4°, we obtain 1·3440 as the sp. gr. of solid nitrobenzene, and 1·2220 as that of the liquid nitrobenzene. The temperature of the liquid nitrobenzene was actually 3·8°, but this I have taken as equal to 4°. I have not attempted to correct the solid nitrobenzene for the increment between 1·5° and 4°, as I have no data for the expansion of the solid substance.

There is thus a contraction of volume amounting to 0·099837, or nearly 1%, as nitrobenzene passes from the liquid to the solid state, whilst conversely, on fusing, the substance expands from 1·000000 to 1·099804.

Temperature of Solidification of Nitrobenzene.

After the specific gravity of the fused mass of crystals had been taken, a thermometer was immersed in the liquid, and a crystal having been dried on filter-paper was introduced, and the whole stirred. It almost immediately commenced to solidify, and the thermometer rose sharply to 5°, at which it remained constant; a thermometer kept in a large mass of the fusing crystals also registered this temperature.

It has been already stated that the liquid obtained from the fused crystals exhibited a slight opalescence or turbidity, due to the presence of moisture condensed from the atmosphere and dissolved by the nitrobenzene to a minute extent. As soon as the crystal was added, and the temperature of the liquid began to rise, the whole became clear, thus indicating in a striking manner the increased power of dissolving water caused by even so small a thermometric rise as from 3·8° to 5°.

Schultz (in *Die Chemie des Steinkohlentheers*, &c., 1882, first edition, p. 355) states, without quoting his authority,

that nitrobenzene has a fusion-point of 3°, a sp. gr. = 1·2002 at 0° and 1·1866 at 4°, no statement as to the temperatures of the water with which comparison is made being given. In the second edition of this work the sp. gr. is stated to be 1·208 at 15°.

Beilstein gives the same numbers, and quotes Mitscherlich as his authority for the fusing-point and Kopp for the specific gravity. Gmelin gives Mitscherlich as authority for both numbers, giving 3° as the fusion-point and 1·209 as the specific gravity. It would appear that no recent observation of these constants has been made.

This year I have repeated the experiments (with the exception of that on the specific gravity of the solid) with a specimen of nitrobenzene prepared from benzene of the highest purity, the nitrobenzene itself being also very carefully purified and twice distilled. The temperature of fusion was obtained with great care by immersing a standard thermometer in a large mass of the solidified nitrobenzene. The thermometer is one made by Casella in 1872; it has a Kew certificate, and has been tested at Kew on two occasions since the date of the original certificate, the last being in 1891. It is a Fahrenheit thermometer, and the correction at 40° to 50° is -2°.

Repeated readings of the former point were taken with this thermometer, and were found to be 43° to 43·1° F.

Several experiments were also made with nitrobenzene obtained by melting well-drained crystals used in the last experiment, cooling below the solidifying point, and introducing the same thermometer, to which some crystals were adhering. The thermometer fell sharply to 41° F., and then the liquid instantly crystallised, the temperature rising as sharply to 43° F., long crystals shooting with extraordinary rapidity through the mass. The number above given, on applying the correction, becomes 41° F. = 5° C.

The specific gravity was 1·2123 at 13° and 1·1974 at 28° compared with water at 15°. Compared with water at 4° these numbers become 1·2116 and 1·1931.

Boiling-point of Nitrobenzene.

Gmelin, quoting Mitscherlich, gives the boiling-point of nitrobenzene as 213°, whilst Schultz (*loc. cit.*, 1882), in his first edition, gives 210°, and in his second (1886) 206° to 207°. Beilstein quotes Städeler as giving 205° at 730 m.m.

The sample was boiled on fragments of platinum foil in one of the pear-shaped flasks used for determining the boiling-points of the aromatic hydrocarbons. A Geissler normal thermometer was used. When the boiling had become steady, the readings were 203·4°, 203·4°, 203·5°, 203·5°, 203·5°, and the liquid was distilled to dryness without further rise. The barometer stood at 755 m.m., or 29·73 inches. Correcting for 760 m.m., the boiling-point 203·5° becomes 203·77°.

The boiling-point, when the mercurial column was totally immersed in the boiling vapour, was 209·8°, 209·9°, 209·8°, 209·9°, 210·0°, 209·9°, pressure being 756 m.m. Taking 209·9°, and correcting for barometer at 760 m.m., this becomes 210·2°. The distillation was not continued to dryness, as the previous experiment had proved the liquid to be homogeneous.

The Geissler thermometer had been in my possession for many years; but having noted the fact that the correction for the Fahrenheit thermometer had changed from +0·1° to -2·0° in twenty years, the zero point of the Geissler thermometer was re-determined and found to be +1°. This corresponds with 1·8° F. It is interesting that two thermometers repeatedly used for liquids boiling near 200° C. should exhibit so very similar an alteration in the zero point.

If this correction is applied, the boiling-points become uncorrected for immersion of column 202·8°. With column immersed, 209·2°.

Summary of Results.

The specific gravity of solid nitrobenzene at 15°/4° is

13440, whilst for liquid nitrobenzene it is $d_{38^\circ/4^\circ}$, 1.2220; $d_{13^\circ/4^\circ}$, 1.2116; $d_{28^\circ/4^\circ}$, 1.1931.

Expansion on fusion, 0.099804.

Contraction on solidification, 0.099837.

Boiling-point at 760 m.m., 209° corrected (that is, with column immersed in the vapour).

Nitrobenzene is remarkable as giving a strongly-coloured vapour. The colour, which resembles that of diluted chlorine, is visible in a layer of 2 inches, and is very marked in a layer of 6 or 8 inches. The vapour does not exhibit a banded spectrum when examined by transmitted light.—*Transactions of the Chemical Society*, 1897.

REMARKS ON THE PART PLAYED BY CHEMISTRY IN PERFUMERY.

By LOUIS OLIVIER.

THE fears expressed by M. J. Rouché on the subject of the introduction of chemistry into the perfumery industry suggest a remark that he would certainly not wish me to express. It is only natural that French perfumery, endowed by nature with a kind of monopoly, fears seeing itself robbed of this monopoly, and that through the progress of chemistry. But this evolution is, if I mistake not, a law of Nature. As it spreads through civilisation, science, an international possession, tends to destroy, for the good of the many, the privileges of the few. It is to the multitude of consumers that it holds out its blessings, and thanks to them a larger number of human beings are enabled to enjoy, and it is continually lowering, the selling price.

Whether we rejoice or are sorry, there is the law. Should we therefore on this account, simply with the view of postponing the end of a privilege, turn our backs on this science, in which our foreign rivals see their safety, and which will permit them to beat us in our own products?

To try and avoid an inevitable evolution, should the French perfumery industry go on rather to a revolution, which would be fatal to it? Instead of letting Germans, English, and Americans make artificial products, of mediocre value perhaps, but of large sale, and thus, to the detriment of the French, take possession of the International market, would it not be better to put oneself in the van of progress, direct it, and assure oneself, independently of the present market, of the acquisition of new ones?

A chemical laboratory and good chemists doubtless represent a serious expense, the more difficult to bear inasmuch as in scientific research there is always the risk of not being immediately successful. But in commerce sacrifices are necessary; we must learn to prepare for the future. If French perfumery decides to have a merry life, it will undoubtedly run the risk of having a short one.—*Revue Générale des Sciences*, 8th year, No. 16, Aug. 30, 1897.

ON THE ESTIMATION OF OXIDE OF IRON AND ALUMINA IN PHOSPHATES.

By N. BLATTNER and J. BRASSEUR.

THE following methods have been studied in the laboratory of the Kuhlmann works at Loos:—

1. The acetic method, or, to be more exact, the acetic acid and ammonia method, carried out in the manner proposed by Maret and Delatree.

2. Glaser's method, based on the preliminary separation of the lime.

3. Lasne's method, by separating the alumina in the

state of phosphate, by means of caustic soda, hyposulphite, and acetate of ammonium.

4. Gruber's method, analogous to the preceding one.

5. Gladding's method, based on the same principle, but making use of caustic potash instead of soda.

6. Thomson's method, by the direct precipitation of phosphate of iron and alumina with ammonia.

MM. Blattner and Brasseur have been led to the following conclusions:—

1. Acetic method. This should be disused: the figures found for alumina are nearly always much too low, the acetic liquid keeping in solution a sensibly constant quantity of alumina.

2. Glaser's method (the alcoholic) gives sufficiently accurate results when the phosphates are free from manganese; it is rapid and easily performed.

3. The caustic soda method gives results scientifically exact, when we observe all the details of the process such as they were described by H. Lasne (*Bull. Soc. Chim.*, xv., p. 118).

4. Gruber's method (described in the *Zeitsch. f. Ang. Chemie*, p. 741, 1896) is nothing but an abridgment or mutilation of Lasne's. It gives inexact results, and should be rejected without hesitation.

5. The caustic potash method, published by Gladding (*Journal of the American Chemical Society*, No. 8, 1896), is analogous to that of Lasne, from which it differs only by modifications in the details of procedure, which might become sources of error.

6. The method of direct precipitation, sufficiently difficult to obtain complete neutralisation (which is indispensable), gives varying results, according to the nature of the phosphate, and the precipitate always contains lime.—*Bull. Soc. Chim.*, Series 3, vol. xvii.-xviii., No. 15.

THE DIRECT SEPARATION AND THE QUANTITATIVE DETERMINATION OF CHLORINE, BROMINE, AND IODINE IN ORGANIC SUBSTANCES.

By P. JANNASCH and E. KOLITZ.

FOR this purpose the specimen is opened up either by the method of Carius or by combustion with caustic lime.

The method of Carius, especially if the quantity of substance is but small, cannot be carried out as quickly and safely as the decomposition in the lime tube.

The further treatment of the melt is the same as in the following lime method:—

For its execution we use potash glass tubes of 50 c.m. in length and 4 m.m. interior diameter, and charge them as follows:—The tube is first filled by means of a tube-funnel, to the depth of 3 to 4 c.m., with quicklime; then the weighed substance loosely ground up in a tall porcelain mortar is added; the mortar, funnel, and tube are then repeatedly rinsed out with finely-ground quicklime, with which 47 c.m. of the tube are filled. The tube is then closed with a loose plug of asbestos, through which a channel, not too narrow, is formed from end to end by tapping it from side to side.

After the tube has become cool again, the reacting mixture is transferred into a litre flask capable of being closed with a ground stopper, and filled to one-third with water. The tube is then rinsed out, at first with water alone and finally with dilute nitric acid.

With continuous shaking and repeated refrigeration, strong nitric acid is added by portions until there is left only a small residue of undissolved caustic lime, and filtered off from this and the liberated carbon. The caustic soda and residual carbon are well washed out with hot water; the liquid on standing (before filtration) must appear absolutely colourless, and the air in the flask must be inodorous. The filter-paper during filtration must not assume a bluish tint.

The silver-haloid, collected and washed, is introduced whilst still wet along with the paper into a silver crucible, and fused with 5 to 6 grms. of chemically pure soda, so as to form a clear quiet liquid.

When the crucible has become cold the melt is taken up with water, preferably on the water-bath.

The caustic lime resulting was found, in every experiment, perfectly free from halogens.—*Zeit. Anorg. Chemie*, xv., p. 68.

SEPARATION OF CHLORINE AND BROMINE IN PRESENCE OF ACETATES, SULPHATES, AND NITRATES.

By P. JANNASCH and E. KOLITZ.

As regards the direct quantitative separation of chlorine and bromine by permanganate in a strong acetic solution, the attempt was made to effect it in presence of large quantities of sodium acetate. The object in view was to raise the boiling-point by the presence of the acetate, and in this manner to facilitate the liberation of the bromine. But it appeared that this purpose was not only not effected, but that the quantitative liberation of the bromine was actually hindered by the addition of acetates. In a series of such attempted separations only very small quantities of bromine distilled over, whilst the main quantity remained behind. Analogous experiments were made with additions of sodium sulphate and sodium nitrate, with results showing that the presence of each is favourable to the reaction, and does not in any manner interfere with the accuracy of the determination of the two halogens then jointly present.

From the observations of the authors and of Azoff, it may be concluded in general that in presence of alkaline fluids the latter may be neutralised or slightly acidulated with sulphuric or nitric acid, but never with acetic acid.—*Zeit. Anorg. Chemie*, xv., p. 66.

A FLAVOUR-PRODUCING MICROCOCCUS OF BUTTER.

By SIMEON C. KEITH, Jun., S.E.

IN April, 1896, I was studying the effects of various bacteria upon cream, and in the course of my experiments I isolated from a mixture of bacteria growing in an agar tube a micrococcus that was found to produce a decided butter flavour and aroma when grown in milk or cream. This proved to be a new species, for which I propose the name *Micrococcus butyri-aromafaciens*.

It has always been the custom to allow cream to sour or "ripen" before churning it for butter, because after this process the butter comes better and more quickly, is of better texture and flavour, and keeps better than butter made from sweet cream. Lord Lister and Pasteur, many years ago, showed that the souring of milk and cream is due to a process of fermentation during which the milk sugar is converted into lactic acid, and that this is due to the activity of minute micro-organisms. It remained for Professor Wilhelm Storch, of Copenhagen, however, to introduce the use of pure cultures of milk-souring bacteria in butter making. Storch found that several kinds of acid-producing bacteria are concerned in the normal souring of cream, and he isolated three species that impart especially fine flavours to butter under favourable conditions.

A similar line of work was taken up by Professor Weigmann, of the Agricultural Experiment Station at Kiel, in Germany, and by Professor H. W. Conn, of Wesleyan University in the United States.

Of the bacteria that have been described as producing a beneficial effect in the ripening of cream, *Micrococcus*

butyri-aromafaciens most nearly resembles Conn's *Bacillus* No. 41 (Storrs Agricultural Experiment Station, *Bulletin* 12, and *Report* for 1894) in its effects upon milk, but it differs in its morphological and in many of its physiological characters. It is a micrococcus growing at 37° and 20° C. It liquefies gelatin slowly, and does not grow well on potato. It may be noted in this connection, however, that recent cultures on gelatin seem to show that the organism has lost to a considerable extent its power to liquefy gelatin during a year's cultivation in the laboratory.

The culture of the micrococcus for use in creameries is propagated in bouillon in Fernbach flasks (broad flasks so constructed that a large surface of liquid is presented to the air). When ready for shipment, the culture is transferred to sterilised bottles under aseptic conditions and hermetically sealed by means of sterilised corks and melted paraffin. Put up in this way, the culture may be kept for an indefinite time without danger of infection by any other organism, but in the sealed bottles the micrococcus loses its vitality so rapidly that after eight days it will no longer produce the best results. Experiments made on a commercial scale show that cream ripened with the aid of fresh, pure cultures of this organism produces generally better butter than the same cream ripened in the usual way. The distinguishing characters of the species are given in the following systematic description.

Micrococcus butyri-aromafaciens, Nov. Sp.

OCCURRENCE.

Isolated from a mixed culture growing on agar in April, 1896.

GENERAL CHARACTERS.

Shape and Arrangement.—A micrococcus occurring generally in pairs.

Size.—0.5 to 0.7 μ in diameter, occasionally reaching 1 μ .

Motility.—Non-motile.

Spore Formation.—No spores.

Relation to Temperature.—Grows rapidly at 37° and 20° C.

Relation to Air.—Aërobic.

Relation to Gelatin.—Slow liquefier.

Colour.—Non-chromogenic (white).

Stain.—Stains well with carbol-fuchsin.

GELATIN.

Stick Culture.—Five days. The gelatin is liquefied in the form of a deep cup $\frac{1}{2}$ inch in diameter. The liquefied gelatin remains clear, with a white film and sediment. The growth below the point of liquefaction is a moderately thick white dotted line.

Plate Culture.

Surface colonies: The colony first appears as a white raised dot which soon sinks in a pit of liquefied gelatin, and ultimately becomes surrounded by a slight whitish ring along edge of the liquefied gelatin.

Submerged colonies: The submerged colonies occur as smooth spherical dots.

AGAR.

Streak Culture.—A very white, smooth, shining growth, which is fairly abundant. The growth is of equal thickness throughout.

Plate Culture.—Characters of no diagnostic value.

Lactose-litmus-agar.—Litmus reddened slightly.

POTATO.

There is very little growth on potato. In two weeks it appears as a very thin white line, barely visible.

MILK.

Not coagulated. A slightly sourish, pleasantly aromatic "buttery" flavour. Slightly acid.

SMITH SOLUTION.

No gas produced. The growth occurs mostly in the open limb of the fermentation tube, the bouillon of the closed limb being only very faintly turbid.

NITRATE.

Reduced to nitrite. Recent cultures do not seem to give this reaction very strongly, although when first isolated it was very marked.

BOUILLON.

Two days, 25° C. Very cloudy with sediment. One week, no further change.

Two days, 37° C. Very cloudy with sediment and ring growth on tube at surface of the liquid.

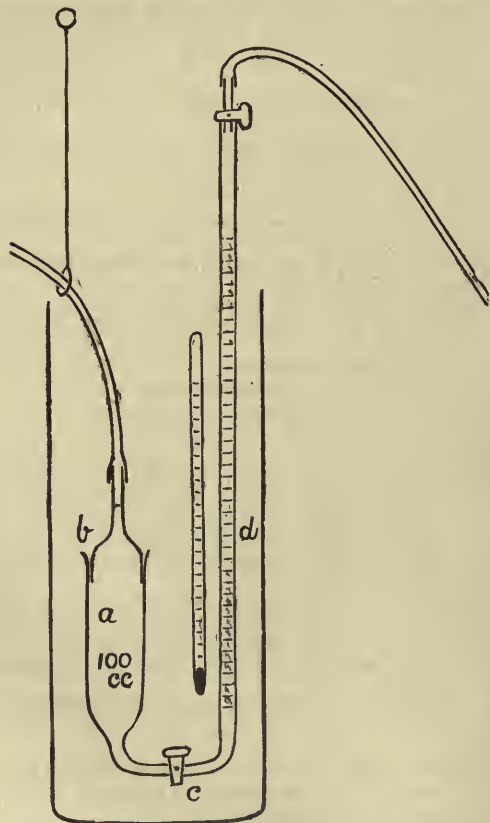
—*Technology Quarterly*, vol. x., No. 2.

SIMPLE LECTURE APPARATUS.

By Prof. W. R. HODGKINSON, Ph.D., F.R.S.E.

VOLUMETER.

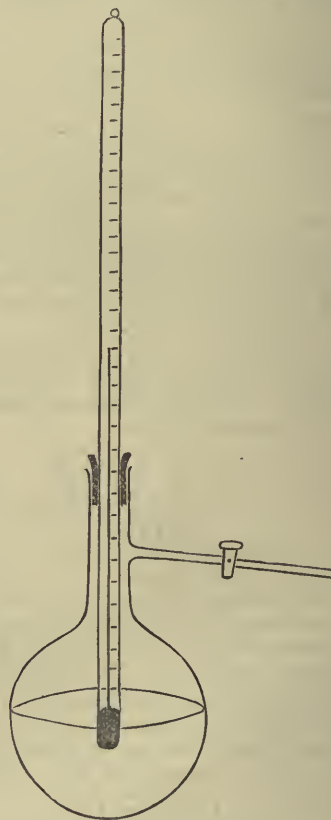
THE apparatus here figured was constructed from the absorption tube of an Orsat-Muencke's gas-analysis apparatus, which had broken at the bend, and an ordinary 50 c.c. burette, the two being joined by sealing a short stopcock tube between (c).



Evidently any convenient liquid may be used for a relative weight or specific gravity determination. Sufficient liquid is introduced to exactly fill the wide tube to the mark *b* and to stand at a moderately low mark on the c.c. tube (*d*), which is read off; the liquid is then lowered from the wide tube to some distance below the stopper, the latter carefully taken out, and allowed to hang by the rubber tube, and the weighed substance (in powder or small pieces) introduced, the stopper replaced, and the liquid carefully brought back to the mark (*b*). The liquid will now stand higher in the graduated tube (*d*); the difference in reading gives the volume in c.c.

The whole apparatus may be sunk in a deep beaker or cylinder of water, which may be retained at any particular temperature during the experiment. The rubber tube attached to the top of the burette serves to apply air pressure to drive the liquid to the mark in the wide tube. This is best done by closing the bottom tap (*c*), blowing strongly into the burette, and closing the top tap. On carefully opening the bottom tap the liquid will rise in the wide tube and the height can be easily adjusted.

The ancient experiment by which is shown that water boils in a closed inverted flask when cold water is poured over it can be somewhat improved upon by employing a distilling or fractionating flask with a stopcock sealed on the side tube and a wide thermometer, wedged by rubber ring in the neck, just dipping into the water. In this case the flask need not be inverted. Other liquids besides water may be used. The temperature can be read off at



which the liquid goes through the process of boiling. The size of flask depends to some extent on the liquid to be employed. For liquids of higher boiling-point than water, a 500 c.c. capacity is enough, and water must not be used for cooling; air from a small bellows is sufficient. For water a 1000 or even 1500 c.c. flask is not too large if the experiment is to be seen at a distance. The mode of working is apparent from the sketch.

Royal Military Academy, Woolwich.

Oxidising Action of α -Monochlorised Camphor.—H. Vittenet. — In the reaction between the aromatic amines and α -monochlorised camphor the authors were surprised to find that, with certain of these bases, colouring-matters were formed which are generally obtained by the direct action of oxidising agents.—*Bull. Soc. Chim. de Paris*, xvii.-xviii., No. 14.

ON THE
PURIFICATION AND ATOMIC WEIGHT
OF CERIUM.*

By MM. WYROUBOFF and VERNEUIL.

(Concluded from p. 139).

Atomic Weight.—Here we come across altogether unexpected difficulties. The sulphate is, up to the present, the only well-crystallised salt of which we can make use, very stable and very easy to purify; now nothing is more difficult than to separate the sulphuric acid from the cerous oxide. Precipitation by oxalate of ammonium (Wolf, Wing) is no good, for oxalate of cerium is not insoluble in acetate of ammonium, and, further, it carries sulphuric acid with it.

The twice-repeated precipitation with soda-lime (Schützenberger) has two drawbacks: if the soda is not in great excess it will not completely decompose the basic sulphate which is formed; in fact, by calcining the oxide of cerium obtained, in a current of hydrogen, we notice very distinctly a more or less considerable disengagement of sulphuretted hydrogen.

If the quantity of soda is sufficient to effect the decomposition, it dissolves cerous oxide, which can be detected by adding a little peroxide of hydrogen. It is true we can separate the cerium and the sulphuric acid integrally in another manner; to do this it suffices to add to the solution an excess of peroxide of hydrogen and caustic soda until the reaction is alkaline, boil to transform the reddish brown peroxide into yellow ceroso-ceric hydroxide. The oxygen thus precipitated and calcined in a current of hydrogen will be found to be perfectly free from sulphur. But this separation does not remove all the difficulties. Modern researches (Ripper, Richards and Parker, &c.) have shown, in the most distinct manner, that the estimation of sulphuric acid in sulphates, in the presence of chlorides, can only be done with a very insufficient approximation, for an operation so delicate as the determination of an atomic weight. We could certainly make the correction for the chlorine entangled by the sulphate of baryta, but Messrs. Parker and Richards have shown that this correction does not always lead to a better result, because of the solubility of barytic sulphate in an acid solution, and further, in the present case we do not know in what condition the entangled chlorine is present. Is it in the state of BaCl_2 or NaCl , or both together? An example will show what variations in the atomic weight can occur according to the method adopted of making the correction. We had in the sulphate of the first fraction of Series I. (see Table), for 100 parts of anhydrous cerous sulphate, 123.384 of SO_4Ba , which gives us the atomic weight—

$$\text{Ce} = 93.01.$$

This sulphate of baryta contained 0.27 per cent of Cl ,† which, counted as NaCl , gives 122.838 of SO_4Ba and an atomic weight of 93.84; counted as BaCl_2 , it gives 122.401 of SO_4Ba and an atomic weight of 94.53. The true atomic weight, as we shall prove further on, is very close to 92.7. We see thus that the method of estimating the sulphuric acid cannot be applied to the determination of the atomic weight of cerium.

M. Brauner made use of another method which at first sight appeared to be quite beyond reproach. He strongly heated the dehydrated sulphate, and calculated the atomic weight from the weight of Ce_2O_3 obtained. The average of his experiments, which were very closely concordant, gave—

$$\text{Ce} = 93.48.$$

Schützenberger however remarked, with very good reason,

that the ceroso-ceric oxide had different weights according to the temperature of calcination, and that therefore one could not tell with accuracy the moment when its composition was really Ce_2O_3 . These variations may attain several thousandths, so he considered that the process could not furnish really exact results.

We can only recall from memory the determinations made by starting with cerous oxalate (Jegel, Bührig, Rammelsberg). We consider this salt as altogether unfit for deciding the atomic weight of cerium; firstly, because it is almost impossible to obtain it in a state of purity, for when it is crystallised it always retains cerous oxalate; and secondly, because organic analyses are subject to errors far too great to allow of their application to so high an atomic weight. The great differences found (Jegel, 91.66; Bührig, 94.4; Rammelsberg, 92.16) show the inefficiency of the method. It is necessary to remark further—these surprises are not rare in the history of cerium—that Bührig's figure 94.4, generally accepted at the present time, is the same as that given by Marignac in 1848, and shortly afterwards, in 1853, admitted by him to be incorrect. He obtained it, as a matter of fact, by the direct precipitation of the sulphate by chloride of barium. Finally, to complete the history, we will cite the analysis of the anhydrous chloride made by Mr. Robinson, which gave—

$$\text{Ce} = 93.5,$$

a figure identical with that obtained by M. Brauner. The chloride is too deliquescent a salt, and too difficult to free from the oxychloride which accompanies it, to allow of its being used for the determination of the atomic weight with any chance of success. All the old methods being rejected as insufficient, there remains now to find a better. We noticed, while making a large number of estimations in water in the hydrated sulphate, that the figures obtained were very closely concordant. On twenty estimations none varied more than 0.03 per cent. Thus the idea occurred to us to employ this estimation for the determination of the atomic weight; in any case it would serve to fix, in an exact manner, the conditions under which the ceroso-ceric oxide, obtained by calcination at a high temperature, would have the formula Ce_2O_3 , and to check with certainty Brauner's method. The table, in which we give a *résumé* of our experiments, shows in a decisive manner:—

1st. That the estimation of the water gives less variations than the ceroso-ceric oxide calcined at a white heat, and consequently a more exact figure for the atomic weight.

2nd. That Brauner's method gives a fairly exact figure when the sulphate is calcined at a very high temperature (about 1500°), because it is only at this high temperature that we can eliminate the last traces of sulphuric acid. In this state, if it is perfectly pure, it should be perfectly white, without the least trace of any rose, chamois, or yellow tint. By this method it is the lowest figures which are nearest the truth.

But to be able to estimate the water accurately, it is of importance which of the numerous hydrated cerous sulphates is used. We therefore commenced by carefully studying the conditions of the formation, and the properties, of these salts, about which there is great want of accord.

The hydrates described are five in number:—

A.	$(\text{SO}_4\text{Ce})^a$	..	..	5 aq.
B.	SO_4Ce	..	..	2 aq.
C.	$(\text{SO}_4\text{Ce})^a$	..	..	8 aq.
D.	SO_4Ce	..	..	3 aq.
E.	SO_4Ce	..	..	4 aq.

The crystalline form and the optical properties of the salts A, C, and D have been determined, at least approximately, by Marignac and Des Cloizeaux. The salt E occurs in an efflorescent form, consisting of a tangled mass of needles quite indeterminate; the salt B only

* Bull. Soc. Chim., Series 3, vol. xvii.-xviii., No. 14.

† This figure is only slightly different to the mean of seventeen analyses made by Messrs. Richards and Parker, who found 0.237 per cent (Zeit. Anal. Chem., viii., p. 417).

TABLE (O = 16; S = 32).

		Hydrated salt.	Anhydrous salt.	Ce ₂ O ₃ .	H ₂ O p.c.	Ce ₂ O ₃ p.c. from		Atomic weight calculated from—		
						Hydrated salt.	Anhydrous salt.	H ₂ O.	Ce ₂ O ₃ from the Hydr. salt.	Ce ₂ O ₃ from the Anhydr. salt.
I.	1.	1·2385	0·9875	0·5977	20·267	48·259	60·526	92·84	93·08	93·16
	2.	1·2730	1·0148	0·6138	20·282	48·216	60·484	92·65	92·88	92·95
	3.	1·2030	0·9590	0·5794	20·282	48·162	60·417	92·65	92·64	92·63
	4.	1·5420	1·2295	0·7430	20·265	48·184	60·431	92·85	92·74	92·70
		Averages ..		20·274	48·205	60·464	92·74	92·84	92·86	
II.	1.	0·9642	0·7685	0·4642	20·296	48·143	60·403	92·49	92·55	92·56
	2.	1·3260	1·0571	0·6380	20·279	48·182	60·438	92·69	92·72	92·73
	3.	1·1429	0·9112	0·5518	20·273	48·280	60·557	92·76	93·17	93·30
	4.	0·9072	0·7232	0·4372	20·282	48·192	60·453	92·66	92·77	92·80
		Averages ..		20·282	48·199	60·463	92·65	92·80	92·85	
III.	1.	1·2114	0·9658	0·5840	20·274	48·208	60·468	92·75	92·84	92·87
	2.	1·2411	0·9894	0·5984	20·280	48·215	60·481	92·68	92·88	92·93
		Averages ..		20·277	48·211	60·474	92·71	92·86	92·90	
Average of the three series ..				20·278	48·205	60·467	92·70	92·83	92·87	
Difference between maxima and minima ..				0·031	0·137	0·154	0·36	0·62	0·74	
Diff. between maxima and the general average ..				0·018	0·075	0·090	0·21	0·34	0·43	

has been vaguely described as crystallising in needles similar to those of the salt A. In reality this salt does not exist, and every time we obtain quantities of water of about 16 per cent we are sure of finding under the microscope two sorts of crystals; prisms of the salt A and octahedra much less bi-refrangible of the salt C.

The conditions under which these diverse hydrates are formed are very different, according to whether the liquid does or does not contain a little free sulphuric acid. We have only here to examine the particular case of solutions absolutely exempt from free acid. This condition is essential, for we know that the cerous sulphate—no matter how well it may be crystallised—retains sulphuric acid with a remarkable tenacity, which may be explained perhaps by the facility with which an acid sulphate, described by one of us some time since (*Bull. Soc. Chim.*, Series 3, vol. ii., p. 745, 1889), is formed, and which is only difficultly decomposed at a relatively very high temperature.

To remove the last traces of sulphuric acid we can proceed in several different ways. We can either precipitate two or three times with alcohol, or, after a preliminary dehydration carried out at as high a temperature as possible (400° to 450°), dissolve, crystallise the greater part at 75° to 80°, evaporate the mother-liquor which retains the greater part of the existing free acid, heat until all sulphuric acid vapours are driven off, dehydrate the crystals, dissolve the whole in water, and repeat the operation three or four times; or again, and in a more simple manner, decompose the nitrates with an insufficient quantity of sulphuric acid, and heat to 500°. The mass contains either ceroso-ceric oxide, or a basic salt which remains on the filter, and cerous sulphate which dissolves. The salt thus prepared gives, at all temperatures up to about 85°, only the hydrate C, sometimes mixed—when the temperature does not go beyond 45°—with a few needle-like crystals, generally isolated and sharply defined, of the hydrate D.* It is only at temperatures of about 100° that we obtain clino-rhombic crystals of the salt A always mixed with the hydrate C. On removal from the mother-liquor these crystals effloresce, or rather appear to effloresce very rapidly; in reality they become covered

with a layer of crystals of the higher hydrate C, which is only normal and stable under these conditions.

It follows from this that the salt (SO₄Ce)₃, 8aq., is the only one which can be used for exact weighings. It keeps very well in the air, easily forms limpid crystals, especially when small. Its density at 17° is 2·885. To obtain it quickly in very limpid crystals it is necessary to dissolve the sulphate, dehydrated at about 400°, in ten times its weight of water, and crystallise at 60°. In twenty-four hours we get an abundant crop of crystals, not more than 2 m.m. in diameter, but perfectly transparent. These are drained on filter-paper until quite dry; they are then powdered and dried anew. In this state they are ready for analysis, and give constant results, even after having been exposed to the air for two or three days. The hydrate C, like all the others, is easily dehydrated at about 250°; if then heated to redness in a tube it will not give a trace of moisture. Once dehydrated, it will stand a temperature of 500° without undergoing the slightest decomposition. On these two points we are in complete discord with M. Brauner, who maintains that the water cannot be entirely driven off even at 400°, and that a temperature of 500° turns the salt yellow by oxidation commencing to take place. We would remark that if our estimation of the water was too low, the atomic weight of cerium—already lower than that of M. Brauner—would be still further reduced; on the other hand, the oxidation of cerous sulphate at 500° clearly shows, as Nilson first observed (*Ann. Chim. Phys.*, Series 5, vol. xxx., p. 431, 1883), the presence of thorium.

If the dehydrated salt is heated to about 1500° it only loses its acid very slowly, and it requires to be kept at this high temperature for at least fifteen minutes before its weight remains constant. The oxide thus obtained appears to be free from sulphur, at least that in the analysis I. 2 (see Table), calcined in hydrogen, did not show any trace. However, the extreme difficulty with which this salt parts with its acid by calcination ought to make us consider this method as less exact than that by estimating the water.

To obtain fairly correct results it is necessary to do the calcination in double crucibles, using platinum crucibles as small as possible, not taking more than 1·5 grms. of material, and weighing in an atmosphere free from moisture.

The three series of analyses given in the table were performed on three products, essentially different:—

* These crystals are at once distinguished under the microscope from the needles of the hydrate A, with which they might be confounded. They belong to the hexagonal system, and in polarised light disappear in the direction of their length; the extinction of the crystals A is, on the contrary, very oblique on the face *m m*.

I. Cerium obtained from rough oxalates from monazite, by the process we have described above, and carefully purified from thorium.—This cerium was transformed into sulphate, and the sulphate fractionated in nine portions. The analyses 1 and 2 were done on the first portion, the analyses 3 and 4 on the last.

II. Cerium obtained from the industrial treatment of oxalates extracted from monazite, very rich in thorina (about 50 per cent) by carbonate of ammonium.—This reagent dissolves a certain quantity of cerium, the didymium, and all the yttria earths. After eliminating the great part of the thorina by the usual methods, the remaining mixture was treated the same as the cerium I. The sulphate gave three fractions. The analyses 1 and 2 are of the first, the analyses 3 and 4 of the last.

III. Cerium extracted from rough oxalates obtained from cerite, and purified like I. and II.—The sulphate was separated by three crystallisations at 60°. The analysis 1 is of the first, the analysis 2 of the last.

The examination of this table shows in the clearest manner that cerium, no matter what its origin, properly freed from impurities and especially from thorium, is an element, pure and simple, giving always a perfectly white oxide, Ce_2O_3 , at a high temperature. The indirect methods which have been used do not allow of its atomic weight being definitely determined, but we can say with certainty that it is close to 92.7, with an approximation of 0.2 to 0.3 per cent, rather less than more.

Having now proved the identity of this element, we propose to study its principal combinations, more especially its numerous oxides.

THE ATOMIC MASS OF TUNGSTEN.*

By WILLETT LEPLEY HARDIN.

(Continued from p. 39).

Preparation of Tungsten Trioxide.

THE material used in the first few series of determinations was obtained from wolframite from Zinnwald, Bohemia. The greenish yellow oxide obtained by digesting this mineral for several days with aqua regia was washed with distilled water and afterwards dissolved in ammonium hydroxide. The solution was evaporated to crystallisation, and the ammonium tungstate which separated out was strongly ignited. The resulting oxide was again dissolved in ammonium hydroxide, the solution was evaporated to crystallisation, and the resulting ammonium tungstate strongly ignited. The oxide thus obtained was placed in a porcelain boat in a combustion tube and gently heated in a current of hydrochloric acid gas to remove the last traces of molybdenum. The material was then re-ignited and placed in a large porcelain dish filled with distilled water. Ammonia gas was conducted into the water for several days, after which the supernatant liquid was syphoned off and evaporated to crystallisation. The ammonium tungstate which separated out was ignited and the process repeated. The material obtained from the second crystallisation was used in the first series of experiments.

Reduction Series.

Tungsten trioxide obtained by the method just described was used in these experiments. The reductions were made in a hard glass combustion-tube in a current of hydrogen, which was first conducted through solutions of ammoniacal silver nitrate, potassium permanganate, alkaline lead nitrate, caustic potash, and finally through sulphuric acid and a tube containing anhydrous calcium chloride. The reduction in each case was continued for several hours at a temperature almost high enough to melt the glass tube. The porcelain boat which contained

the oxide was protected from the glass tube by means of platinum foil. The weighings were made on a Troemner short-armed balance with a set of weights which had been previously calibrated. The balance is sensitive to the fortieth of a m.grm. The results, calculated on the basis of O = 16, are as follows:—

	Weight of WO_3 . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	1.64084	1.30100	184.05
2	1.79728	1.42550	184.044
3	2.60739	2.06788	183.98
4	4.57390	3.62890	184.33

At this point an unglazed porcelain tube was substituted for the glass tube, and the reductions that follow were continued for three hours at the highest temperature obtainable in a combustion furnace.

	Weight of WO_3 . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	3.32320	2.63547	183.94
2	6.11056	4.84580	183.91
3	9.23802	7.32393	183.66

The last experiment was continued through a period of eight hours.

The first three results agree very closely and give 184.02 as a mean for the atomic mass of tungsten. The mean of the results with the glass tube is 184.10. The maximum deviation is 0.35. The mean of the results obtained with the porcelain tube is 183.84, with a maximum difference of 0.28. The maximum deviation in the whole series is 0.67.

Oxidation Series.

The metal obtained in the foregoing reductions was used in these experiments. The oxidations were made in porcelain crucibles. The material was protected from particles of dust by means of a porcelain dish suspended a short distance above the crucible. The oxidation in each case was continued until there was no further increase in weight.

	Weight of W. Grms.	Weight of WO_3 . Grms.	Atomic mass of tungsten.
1	1.70220	2.14400	184.94
2	1.37651	1.73393	184.86
3	2.05606	2.58951	185.00
4	1.10300	1.38933	184.91
5	1.85855	2.34143	184.75
6	7.28774	9.18730	184.15

The mean of the first five results of this series is 184.89. This value is almost identical with that obtained by Pennington and Smith. The mean of the whole series is 184.77. The maximum deviation is 0.85.

Reduction of Oxide obtained by the Ignition of Metal.

Inasmuch as the value obtained in the oxidation series is almost a unit greater than that obtained by reduction, it was thought advisable to make a series of reductions of the oxide obtained in the series of oxidations.

	Weight of WO_3 . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	2.02890	1.61071	184.88
2	2.15894	1.71388	184.85
3	2.35206	1.86740	184.94
4	1.39137	1.10351	184.01
5	1.92125	1.52487	184.66
6	1.46746	1.16383	183.99
7	5.01313	3.97560	183.93
8	6.11056	4.84580	183.91

The first three results of this series agree very closely and give 184.89 as a mean for the atomic mass of tungsten. The last three results are equally concordant and give 183.94 as the mean value. The mean of the whole series is 184.40. The maximum deviation is 1.03. The oxide used in these experiments was very light and fluffy.

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xix., No. 8.

The material used in the last experiment was moistened and re-ignited to render it more compact.

Oxidation of Metal obtained from the Second Reduction.

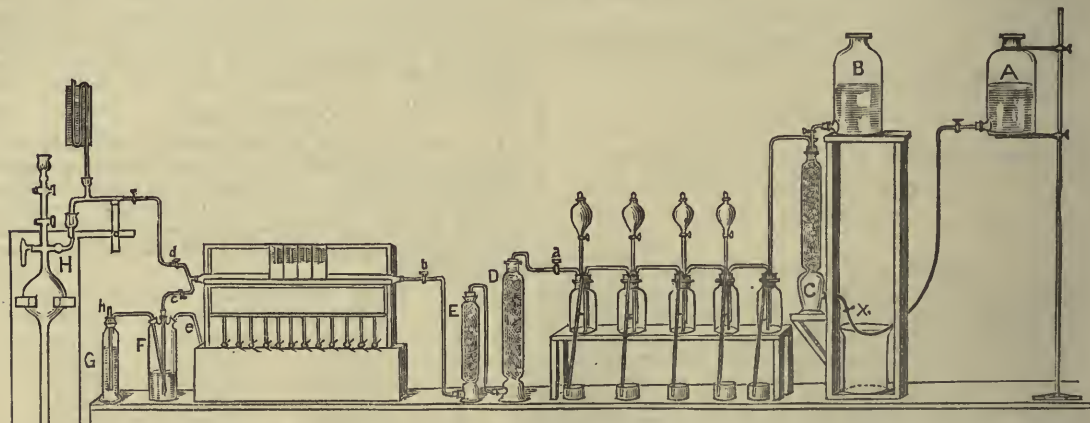
The material obtained in the last series was used in these experiments.

	Weight of W. Grms.	Weight of WO_3 . Grms.	Atomic mass of tungsten.
1	3.96360	4.99460	184.53
2	2.63034	3.31647	184.01
3	1.60964	2.02804	184.65

The variations in this series are similar in every respect to those of the preceding series.

In view of the wide variations in all the preceding work, an attempt was made to weigh the water formed in the reduction of tungsten trioxide. The moisture was collected in a small glass-stoppered U-tube filled with anhydrous calcium chloride. To this tube was attached another similar tube to prevent absorption of moisture from the air. Several determinations were made, but the results were too discordant to establish anything. The removal of the last traces of air from the generator and wash-bottles was very difficult to accomplish, and hence the results were usually too high. The presence of air, even in small quantities, would also affect the results in

The stopcocks of the different separatory funnels were then opened and the solutions allowed to pass into the corresponding wash-bottles. The first bottle contained pure water, the second ammoniacal silver nitrate, the third and fourth potassium permanganate, and the fifth alkaline lead nitrate. The drying tower D was filled with anhydrous calcium chloride and caustic potash, and the tower E with alternate layers of glass-wool and phosphorus pentoxide. These were substituted for sulphuric acid, for, according to Dittmar and Henderson (*Proc. Phil. Soc., Glasgow*), hydrogen when passed through sulphuric acid becomes contaminated, owing to the reduction of the acid by the hydrogen. From the drying-towers the hydrogen passed into a thin-walled glazed porcelain tube, placed in a combustion furnace. The bottles G and F acted as a regulator; the outlet to F was connected to a suction tube, *e*. When the suction was greater than the backward pressure of the wash-bottles, air passed in at *h* and through the columns of sulphuric acid in G and F. The length of these two columns of acid were adjusted so that the pressure exerted against the air passing through them was equal to the backward pressure of the wash-bottles. The Sprengel pump, H, was attached so that the metal might be cooled in a vacuum and thus prevent any occlusion of hydrogen. When the reductions were complete the stopcocks at *b* and *c* were closed. The reservoir



the reduction series. To overcome this difficulty, a different form of apparatus was constructed, the plan of which is shown in the accompanying sketch.

At the beginning of the operation, water, which had recently boiled, was allowed to pass from the bottle, A, into the tower, c, containing granulated zinc. When c was completely filled, the water passed through the small outlet tube into the first wash-bottle; after filling this it passed into the second, and so on. When the water reached the bottom of the cork in the last wash-bottle, the stop-cock at *a* was closed. The stopcock in each of the separatory funnels was then opened and the water allowed to rise until the stems of the funnels were completely filled. In this way the air in the generator and wash-bottles was completely displaced by water. The stopcock at the bottle A was now closed and sulphuric acid allowed to drop from the bottle B upon the zinc in c, the clip at *x* being opened at the same time. The hydrogen formed by the sulphuric acid and zinc forced the water out of c into the beaker below, after which the clip at *x* was closed and the clip on the syphon of the first wash-bottle opened. When the water was removed from this bottle, the clip was closed and that of the second syphon opened, and so on until the water in all these bottles was displaced by hydrogen. The separatory funnels were then filled with the different solutions used in purifying the hydrogen. The stopcocks at *a*, *b*, and *c* were opened, while that at *d* was closed; this allowed the hydrogen to pass through the apparatus.

of the vacuum pump was exhausted and the stopcock at *d* opened. This was repeated several times, until the vacuum was almost perfect.

This form of apparatus was used in all the reductions which followed. The air could be completely removed from the apparatus in a short time. The reductions were continued for a period of three hours at the highest temperature obtainable in a combustion furnace. When the quantity of material exceeded 3 grms. the time was longer.

Reduction Series.

All the material resulting from the preceding experiments was ignited and digested for several days with pure aqueous ammonia. A residue was left which gave the bead test for silica. It is evident from this, that tungstic acid, when reduced in a porcelain boat, takes up silica. The solution of ammonium tungstate was syphoned off and evaporated to crystallisation. The crystals of ammonium tungstate were strongly ignited, and the resulting oxide used in the experiments. For the first time the metal was allowed to cool in a vacuum.

	Weight of WO_3 . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	3.55192	2.81560	183.55
2	4.59362	3.64461	184.34
3	4.30435	3.41459	184.21
4	2.64671	2.09900	183.95

The mean of these four results is 184.01. The maximum difference is 0.79.

Oxidation Series.

The metal obtained in the preceding series of results was re-oxidised, and the following values obtained for the atomic mass of tungsten:—

	Weight of W. Grms.	Weight of WO ₃ . Grms.	Atomic mass of tungsten.
1	2.80958	3.54370	183.70
2	3.63095	4.57662	184.30
3	2.09740	2.64455	183.99

This series, like the preceding, is of little value, owing to the wide variation in the results.

Inasmuch as the removal of air from the present form of apparatus was a matter of little difficulty, another attempt was made to collect the water formed in the reduction of tungsten trioxide, and from its weight calculate the atomic mass of tungsten. The moisture was collected in a glass-stoppered U-tube filled with alternate layers of glass-wool and phosphorus pentoxide. From a series of blank experiments, it seemed that any error introduced by the presence of air in the apparatus would be almost inappreciable. The following results were obtained:—

	Weight of WO ₃ . Grms.	Weight of H ₂ O. Grms.	Atomic mass of tungsten.
1	5.01313	1.16742	184.07
2	2.02890	0.47090	184.86
3	7.04192	1.63864	184.27
4	3.34204	0.77832	184.07

The variations in this series are similar in every respect to those of the different reduction and oxidation series.

(To be continued).

NOTICES OF BOOKS.

The Stamp Milling of Gold Ores. By T. A. RICKARD, M.E., F.R.G.S., A.R.S.M. New York and London: The Scientific Publishing Co. 1897. Pp. 260.

PROBABLY the most important point in gold-winning, and one that in most cases is entirely neglected, is deciding on the best and most economical method of extracting the gold from the ore, after it is brought to bank. Hundreds of thousands of pounds have been lost and wasted, through the ignorance of those in charge of the mine, on this vital question.

A new gold-field is discovered; there is a rush; mushroom experts spring up, who give reports on properties (50 to 500 guineas, according to the proposed capital); companies are floated, operations commenced, and when a certain quantity of ore is being raised the Directors (who as a rule know nothing whatever about the subject) decide to erect a mill; they do so, and very good mills they are, but in ninety cases out of a hundred they are quite unfit for the work for which they have been bought, and, though assays show a payable amount of gold in the quartz, the mill cannot extract it, and about 60 per cent goes away in the tailings. Now this enormous loss could be avoided if only the Directors would consult a known practical man, with a reputation to keep, who could advise them whether their particular grade of ore was more adapted to fast or slow stamping, long or short drop, deep or shallow boxes, inside or outside plates, and all those other *little* details of construction and working the neglect of which leads to 60 per cent loss (a very common figure in the Transvaal).

All these points, and many others, are clearly and thoroughly discussed in these pages.

In Chapter I., the Philosophy of the Stamp-milling Process, the author gives examples of two methods of milling very wide apart in their details, but each well adapted to the class of ore to be dealt with. In Colorado the ore is of complex pyritic character, containing 15 per cent of sulphides, and the gold is in a very fine state of

division; it is therefore necessary to have a long 20-inch drop of about thirty to the minute, a deep box of about 14 inches discharge, and inside plates; on the other hand, in California, where there is only about 1 per cent of sulphides present in the ore, this method would be utterly inadequate; so in that district they use a 5- or 6-inch drop of 100 or 105 per minute, a shallow box, coarser screens, and outside plates only: both these methods, which are now thoroughly understood, sprang from a common origin between the two extremes, and were taken west by the miners from Georgia, but experience and observation has modified them in the manner indicated, to meet the necessities of the ore dealt with.

In succeeding chapters the author describes the methods of milling adopted, after long and costly experience, in all parts of the world:—the early Australian methods, as used at the Clunes, Port Philip, and Colonial Mining Companies; the more modern Australian methods, used at Ballarat, Star in the East, Britannia United, &c.; the Stamp Mills of Otago, New Zealand; and in Chap. XIII. we come to a Review of Australian Processes. In this chapter the relative merits of wet *versus* dry processes are discussed, and Mr. Rickard seems to be of the opinion that the adoption of one or the other is largely dependent on the proximity of the gold-fields to the railway system. In America, as the railway extends, there is a tendency for the smelter to replace the millman, for fire reduction processes to supplant wet methods of gold extraction. In Australia, as yet, the smelter is only to be found in the silver regions; elsewhere the stamp-mill is supreme.

Chapters on Mills and Millmen, and the Future of the Stamp-mill, will be found very interesting reading, and they bring to a close an excellent practical work by a man who shows throughout that he is a thorough master of his subject, and, from a practical point of view, we can only add that we wish there were many more like him.

Bulletin of the Agricultural Experiment Station, Baton Rouge. By D. N. BARROW, Assistant-Director. Issued by the Bureau of Agriculture and Immigration. Series 2, No. 47. 1897.

THE experiments described in this Bulletin are on Corn, Cotton, Forage Crops, and Tobacco. The dry weather has materially cut down the yields, by depriving the crops of the moisture necessary for taking up the available plant food, with the result that all the results have been completely vitiated from an experimental standpoint. Still the yields of cotton and corn were very fair, showing the advantage to be gained by a thorough preparation of the soil and frequent cultivation.

The experiments with corn were a repetition of those made last year. The yields afforded by twenty different varieties are here given, in connection with fertiliser tests. The highest yield per acre was of Farmer's Pride, viz., 35.3 bushels; the lowest being Improved Leoming, which only gave 13.5 bushels per acre. The fertiliser tests with corn are still uncertain and unsatisfactory, owing to the continued recurrence of dry weather for the past eight years just at the time when moisture was most needed. Phosphoric acid in its various forms has always been seen to be beneficial, but there is no definite evidence in favour of one form or another.

The work on the station with regard to cotton has, since the introduction of tobacco, been confined to variety tests, and the results are given in a table. The short staples were ginned and the percentage of lint to seed estimated; the long staples are awaiting the arrival of a roller gin from Europe.

Twelve varieties of forage plants were experimented on, the best results being obtained from *Desmodium molle*, which yielded 42,429 lbs. per acre; the lowest was of *Teosinte*, which gave only 6120 lbs. per acre.

The experiments with varieties of tobacco were carried out on Plot No. 12, which was formerly devoted to nitrogen experiments on cotton. The rows were 3½ feet wide,

and ran at right angles to the old nitrogen rows. Twenty-two varieties were set, but they all suffered so severely from the dry weather that the results are practically worthless—hence the tabulated results convey no information of any value. In the experiments on fertilisers and tobacco it is singular that, though emphatically a "potash-loving" plant, there is no indication of that substance having benefitted tobacco on this soil.

Since the publication of No. 41 Bulletin the tobacco raised on the experimental station has been worked up into cigars. The cigars were recently on exhibition at the State Agricultural Society, and were pronounced by experts to be of excellent quality, and it is expected that they will soon (in eighteen months or two years) compare favourably with Havanas.

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THIS Catalogue (No. 80, 1897), in addition to its usual features, contains an account of works on the constants employed in the analysis of fats and oils. There is also a list of monographs and other original memoirs on chemistry not at present to be obtained in any other form. We feel therefore free to call the special attention of students, and also of librarians, &c., to this list.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxv., No. 8, August 23, 1897.

Critical Constants of certain Gases.—A. Leduc and P. Sacerdote.—The critical temperatures are obtained by us at less than about 0.5, and the critical pressures at less than 1 atmosphere.

The Absorption of the X Rays.—Abel Buguet.—To determine the relations existing between the thickness of a body and its opacity for the X rays the author uses scales of thickness.

No. 9, August 30, 1897.

Photography of the Fluoroscopic Image.—Charles Porchier.—To oppose an absolute barrier to the X rays I arrange the experiment as follows:—The door of the dark chamber of the laboratory is perforated with an aperture in which is placed the object-lens of my photographic apparatus. Besides a barrier of lead behind the door and all around the object-glass, in a radius of 0.50 metre, I have nailed a sheet of lead of 0.003 metre in thickness. Thus a barrier of lead, the metal screen, and the object-glass protects the gelatino-bromide film absolutely against the action of the X rays. The fluoroscopic image is the most interfered with, and can act alone upon the plate. The screen is placed at 0.57 metre from the objective, and the focus of the phial is only 0.03 behind it.

Bulletin de la Société Chimique de Paris.

Series 3, Vol. xvii.-xviii., No. 14.

M. Ponsot made some observations relative to the congelation of milk; in his opinion it is not proved that cryoscopy alone can prove the addition of water or determine its extent.

M. Delépine gave the results of his thermo-chemical experiments on formic aldehyd; his conclusions as to its molecular formation conform with those of Tollens and Grossmann.

M. Causse described his experiments on the action of

salicylic aldehyd on urea; when a mixture of these substances is heated to 110° they combine with elimination of water, forming salicyl-triurea.

M. Hébert presented, on behalf of M. Rabaut, his researches made on the combinations of cupreous chloride with nitrites.

A Method of Oxidation and Chloridation.—A. Villiers.—Already noticed.

Destruction of Organic Matters in Toxicology.—A. Villiers.—Already noticed.

Products of Decomposition of Carbide of Calcium by Water.—E. Chuard.—Already noticed.

Purification and Atomic Weight of Cerium.—MM. Wyruboff and Verneuil.—(See pp. 137 and 153).

On Sacchareines, New Colouring-matters derived from Benzoic Sulphimide (Saccharine).—P. Monnet and J. Koetschet.—A long paper, not suitable for abstraction.

Isomerism existing between Pilocarpidine and Pilocarpine.—A. Petit and M. Polonovski.—From their recently-published researches on these bodies, the authors have been led to doubt the accuracy of the formulae they had adopted, and to suspect the existence of isomerism between these two bodies. The experiments tried confirmed their belief.

Alkalimetric Estimation of Metals: Mercury.—H. Lesœur.—Not suitable for abstraction.

MISCELLANEOUS.

South West London Polytechnic Institute.—Principal, Professor Herbert Tomlinson, B.A., F.R.S. Professor of Chemistry, J. B. Coleman, A.R.C.S., F.I.C., F.C.S. This Institute was opened in 1895, and is therefore commencing its third session's work. Two and three year courses of instruction are given in Chemistry and in Mechanical and Electrical Engineering, suited to the requirements of analysts, industrial chemists, engineers, and others. The chemical department contains two laboratories, balance, lecture, and store rooms, and is designed and equipped for advanced chemical work. In addition to the day courses, evening instruction is given in Pure Chemistry, Pharmacy, Photography, Colours, and other branches of Applied Chemistry.

City and Guilds of London Institute.—A special course of instruction in Electro-chemistry will be given during the coming session at the City and Guilds Central Technical College. The course will include practical instruction in electro-deposition, the use of the electric furnace, dynamos, transformers, and accumulators. A great part of the time of the students attending the course will be devoted to Electro-chemical Research and the study of Electro-chemical Action. Candidates for admission will be required to submit evidence of having a general knowledge of physics and chemistry, and of having been specially trained in one of these subjects.

On the Essence of Bitter Fennel.—E. Tardy.—The French essence of bitter fennel was first washed with an aqueous solution of potash, then with distilled water. The washings, treated with hydrochloric acid, gave anisic acid. The washed essence was then treated with bisulphite of soda, when an abundant precipitate was produced. This precipitate, after being thoroughly washed with ether, was decomposed by potash. The oily liquid produced was submitted to fractional precipitation, by which means it was divided into seven portions, the first boiling below 240° and the latter between 265° and 270°. Above this point a product is obtained of a fatty appearance, in which are a large number of crystalline flakes: these can be separated by dissolving them in ether and re-crystallising. This body combines neither with acids nor alkalies, and on analysis gives the formula $C_{13}H_{14}O_2$.—*Bull. Soc. Chim. de Paris*, No. 13, 1897.

Researches on the Active Principles of some Aroides.—A. Hébert and F. Heim.—The authors have arrived at the conclusion that the active principles of the *Arum* and allied plants are more or less rich in saponine, according to the season, but the maximum proportion never exceeds 1 part per thousand of the weight of the living plant; that the acrid principle, which has not yet been properly described nor even extracted by any one, is a liquid alkaloid, very similar to conicine; and that it is impossible to detect hydrocyanic acid in the aroids they have worked on.—*Bull. Soc. Chim. de Paris*, No. 13, 1897.

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THE CHEMICAL NEWS.

Vol. LXXVI., No. 1975.

THE PERMEABILITY OF ELEMENTS OF LOW ATOMIC WEIGHT TO THE RÖNTGEN RAYS.*

By JOHN WADDELL, B.A. (Dal. Coll.), B.Sc. (Lond.), Ph.D. (Heidelberg), D.Sc. (Edin.).

At the meeting of the British Association held last year in Liverpool, a paper by Dr. Gladstone and Mr. Hibbert was read. It was afterwards published in the *CHEMICAL NEWS*, but before I saw their article I had sent for publication, in the same journal, an account of work that I had done along somewhat similar lines, though starting from a different point and undertaken with a different object. My results to a certain extent coincided with theirs, but they were also to a certain extent different, and I wish to bring before the Association some of these differences, as well as some other phenomena connected with the permeability of powders to the Röntgen rays.

My article in the *CHEMICAL NEWS* dealt not only with a number of metals, but with non-metals as well.

In the case of the metals I used mainly oxides and carbonates, whereas Gladstone and Hibbert used formates and acetates. They say that they used the formate because of the low equivalent of the acid radical (CHO_2). As a matter of fact, the carbonate is better from that point of view, as may be seen by considering that 37 parts by weight of lithium carbonate contain as much lithium as 52 parts of the formate, and 100 parts of calcium carbonate contain as much calcium as 130 parts of calcium formate. Of course the disparity between carbonate and acetate is still greater, and equally of course the oxide has a still larger proportion of metal. In any case it is a matter of comparative indifference which of the compounds is taken, since the permeability of carbon, hydrogen, and oxygen is so small.

A point of supreme importance, however, is to define strictly what is meant by saying that one substance is more permeable or has less absorption than another. For instance, Gladstone and Hibbert, in reference to potassium and calcium, say "It was found that, whether tested by the uncombined metals or by their salts, the absorption of the Röntgen rays by the two metals are the same for thicknesses answering to the atomic weights, while they are very different for thicknesses answering to their combining proportions." It all depends on what is meant by "thicknesses answering to the atomic weights," whether the above statement is correct or not. If it means that a plate of potassium 3.9 m.m. thick has the same absorption as a plate of calcium 4.0 m.m. thick, my experiments tend to prove that it is not correct; but if it means that two plates of the same area, and of such a thickness that the weight of potassium would be 0.39 gm. and of calcium 0.40 gm., would absorb equally, I should agree that the statement is within the limits of experimental error. My statement in the *CHEMICAL NEWS* (vol. lxxiv., p. 298) is "The potassium carbonate was of appreciably the same transparency as the calcium carbonate containing the same amount of metal."

I suppose that when the statement was first made that the absorption by metals was proportional to their density, it was intended that equal thicknesses of metal foil should be used. If this law had been true, it would follow that for thicknesses inversely proportional to the density, the absorption would be equal. I believe that this statement

is not very far from correct with regard to the metals of high atomic weight,—say all of those above iron,—but the absorption of all is so large that it is somewhat difficult to establish or to disprove the statement; but there is little difficulty in proving that it does not hold in comparing metals of high atomic weight with those of low atomic weight. Sodium, magnesium, aluminium, are in quite a different class from iron, copper, lead. So much must be admitted by any one who has done work in this connection; but when a comparison is made among the elements of low atomic weight themselves, it seems that there is room for difference of opinion.

I think it must be conceded that the only fair way to test the relative permeability is to have the radiations pass through the same *weight* of the different metals. In order to do this I took pill-boxes of the same size, and put in them such weights of the compounds that they would contain 1.0 gm. or 0.5 gm. of the metal. This was equivalent to taking sheets of metal inversely proportional to their density, and was of course available for many metals which it would be impossible to obtain in sheets in the metallic state. So far as could be determined with metals of high atomic weight, the permeability of equal weights was approximately equal. Tested in this way, however, the permeability of potassium was much less than that of sodium, or magnesium, or aluminium. I found that the difference between lithium and sodium was not nearly so great as between sodium and potassium, and that, if anything, lithium was less permeable than sodium. This is opposed to the statement of Gladstone and Hibbert, who say "that the order of absorption is lithium, sodium, and potassium, while the order of density is lithium, potassium, and sodium." If the weights of lithium and sodium tested, or even the weights of their formates, were in the ratio of 7 : 23, I should agree with them; but that this is an unfair mode of comparison will be apparent, when it is considered that it corresponds to taking for the test a plate of lithium about two-thirds the thickness of the sodium plate; and no one would think of comparing a plate of aluminium with a *thicker* plate of platinum, which would be a similar experiment.

In view of the variance that appears to exist in the results obtained by Gladstone and Hibbert and myself, I have made a careful re-investigation of the matter, and compared more closely not only lithium and sodium, but beryllium and magnesium, and boron and aluminium. The method of procedure was the same as that employed before, namely, protecting the photographic plate by a sheet of lead in which holes were punched for the pill-boxes. The length of exposure to the Röntgen rays differed in the different experiments, and the battery current and length of spark varied when using the same tube, and two different tubes even were employed. I had found, from previous experience, that it is better to test the permeability of substances in this way than to test their absorptive power by leaving the photographic plate exposed to the full power of the rays, except where covered by the material to be tested.

In the new investigation I took two sets of salts of sodium and lithium, namely, the nitrates and carbonates, and I took varying proportions. In the case of the nitrates I took of sodium nitrate 2 grms. and of lithium nitrate 5.4 grms., $3.2 \text{ grms.} = 5.4 \times \frac{1}{2}$, the ratio of the densities, and 2 grms. so as to have equal weights of the two salts: 2 grms. of sodium nitrate and 5.4 grms. of lithium nitrate contain each a little over 0.54 gm. of the metal.

In every photograph the 2 grms. of sodium nitrate was decidedly more permeable than the 5.4 grms. of lithium nitrate, and much more nearly equal to the 3.2 grms. of the lithium salt, which contained only three-fifths as much metal.

I usually considered that the sodium nitrate was more permeable than the 3.2 grms. of lithium nitrate, but the difference was so slight that it was hard to decide. Such

* Read before the British Association (Section B), Toronto Meeting, 1897.

being the case, it naturally follows that the 2 grms. of lithium nitrate was more permeable than the same amount of sodium nitrate.

In the case of the carbonates the weights employed were, of sodium carbonate 1 gm., and of lithium carbonate 2.3 grms., which contained the same weight of metal, namely, 0.43 gm. I had, in addition, two other boxes of lithium carbonate, one containing 1.0 gm., the other 0.7 gm. In this last the weight of lithium is $\frac{3}{4}$ the weight of sodium in the sodium carbonate employed. The difference between the carbonates which contained equal weights of metal was not so marked as in the case of the nitrates—perhaps because of the smaller actual amount of the metal employed; but on all the photographs, except one, which received a short exposure, and which showed several abnormalities, the sodium carbonate was distinctly enough shown to be the more permeable. When the smaller weights of lithium were employed they were more permeable than the sodium carbonate.

Even the 0.7 gm. of lithium carbonate which contains less than 0.14 gm. of lithium had a perceptible absorption, and, if it is considered that this was spread over an area of more than a centimetre diameter, it would appear to be too strong a statement to say that lithium has "next to no absorbent action on the Röntgen rays."

A result that surprised me was that lithium nitrate containing 0.2 gm. of lithium was more absorbent than lithium carbonate containing 0.43 gm. of lithium. The total quantity of lithium nitrate was 2 grms. and of lithium carbonate 2.3 grms.

I therefore tried another experiment in which sodium and lithium nitrate were compared with sodium and lithium carbonate. The salts were taken in such quantities that they contained each 0.27 gm. of metal, that being the amount of sodium in 1 gm. of sodium nitrate. They were placed near the centre of the plate, and placed symmetrically with regard to the Newton's tube; that is, the line joining the cathode and the reflecting anode was in the same plane as the line passing through the middle of the photographic plate, and on each side of this line the nitrates and carbonates were placed. The sodium nitrate was more permeable than the lithium nitrate; there was hardly any appreciable difference between the carbonates, but the sodium was if anything the more permeable. It was no more, however, than might be accounted for by the difference in quantity of the acid radical. The carbonates were very considerably more permeable than the nitrates. I was induced to compare the permeability of substances containing only carbon, hydrogen, oxygen, and nitrogen, with salts of metals. I tried the relative absorption of 1 gm. of dinitrobenzol, 2 grms. of sugar, and 2 of ammonium nitrate. The dinitrobenzol and ammonium nitrate were chosen on account of the considerable quantity of nitrogen in them; in sugar, of course, nitrogen was absent. The sugar was decidedly less permeable than the dinitrobenzol (of which there was only half the amount), and slightly more permeable than the equal quantity of ammonium nitrate. Sodium fluoride and sodium carbonate, containing the same quantity of metal as the ammonium nitrate did of nitrogen, were somewhat less permeable, though the difference was not so great as would, I think, be generally supposed. The sodium fluoride was very slightly less permeable than the sodium carbonate. All of the substances, however, even the 1 gm. of dinitrobenzol, had an appreciable absorbent action.

I also tried potassium and calcium nitrates and carbonates. There was not so much difference between the permeability of the nitrates and carbonates in these cases, partly doubtless because the acid radical is not so large a fraction of the total weight as in the case of lithium and sodium, and mainly that the great absorbent action of potassium and calcium hides that of the carbon, nitrogen, and oxygen.

Since the acid radical has some absorbent action, the greater permeability that I found in the sodium compounds

may be due to their containing a smaller quantity of the acid radical; but one thing is certain, that it is not correct to assert that lithium is of small absorbent action, while indicating that sodium has considerable absorptive power. Though I have not tested it, I should be inclined to expect that lithium chloride containing a gm. of chlorine, which has a very considerable absorptive power, would not show appreciably greater permeability than sodium chloride containing the same amount of chlorine. I am quite ready to admit that, if the permeability of the acid radical of the carbonates is to be tested or compared with that of the nitrates, the lithium salt is better than the sodium salt, because there is less than one-third the quantity of metal in the former as in the latter.

(Note.—Since the rest of the paper was written I have experimented on the absorbent action of the acid radical of the carbonates. I made the comparison with the absorbent action of metallic sodium. For this purpose I took four different quantities of sodium, which I moulded into flat plates to cover the bottom of the pill-boxes. I protected the sodium by a thin coating of vaseline. There were of sodium 0.25 gm., 0.50 gm., 0.75 gm., and 1.0 gm. Of sodium carbonate, I had in another box 1.73 grms., which contained 0.75 gm. of sodium, and of lithium carbonate 1.21 grms., which had the same quantity of the CO_3 radical as the sodium carbonate. The sodium carbonate was less permeable than the 0.75 gm. of sodium, and more permeable than the 1.0 gm., and would I think be about equal to 0.90 gm. of sodium. That would make the permeability of the CO_3 group the same as of 0.15 gm. of sodium, whereas the sodium in the salt is 0.75 gm.—that is, the permeability of the CO_3 group is five times as great as the sodium with which it combines. It was found that the permeability of the lithium carbonate was less than that of 0.25 gm. of sodium, and greater than that of 0.50 gm. of sodium, and would be about equal to 0.40 gm. of sodium. As the CO_3 group has a permeability of 0.15 gm. of sodium, the lithium in the carbonate has a permeability of 0.25 gm. of sodium. In order to compare lithium with an equal weight of sodium, this number must be multiplied by $\frac{4}{3}$, the result being that, if a plate of lithium of the same weight as sodium be compared with it as regards permeability, it is practically equal, being in the ratio of 0.75 to 0.25 $\times \frac{4}{3}$, if the numbers taken above be regarded as correct, as they approximately are. In the same experiment I found that 2 grms. of ammonium nitrate are not so permeable as 0.50 gm. of sodium. This probably accounts for the nitrates being less permeable than the carbonates. The lithium carbonate had a very appreciable absorbent power, the photographic plate being darkened about half as much as when not protected, except by the paper covering and an empty box).

While making the experiments with lithium and sodium I also compared beryllium and magnesium. I used beryllium oxide obtained by heating carbonate which had been obtained from Schuchardt. The magnesium was taken in the form of carbonate. There was 0.53 gm. of the former, and 0.67 gm. of the latter, each containing about 0.19 gm. of the metal.

The difference of permeability between the beryllium compound and the magnesium compound was slight, and in some cases was in the one direction, and in some cases in the other, perhaps depending upon the relative position on the plate or possibly on the length of exposure; but in all instances they were more permeable than the lithium carbonate containing the same amount of metal. It must be remembered that there was a greater quantity of the lithium salt, and though the acid radical has not a very large absorbent power it is not without influence.

Aluminium and boron also were compared, the oxides being the compounds taken: 1 gm. of the aluminium oxide and 1.68 grms. of the boron oxide were employed. The former was obtained by heating ammonium alum, and the latter by heating boric acid.

The aluminium compound was less permeable than

than that of boron, but even it had a greater permeability than the sodium nitrate which contained the same amount of metal (0.54 grm.), and indeed a greater permeability than sodium carbonate containing 0.43 grm. of sodium only.

Boracic acid, which, assuming its formula to be H_3BO_3 , contained the same amount of boron as the aluminium oxide did of aluminium, was, however, not more permeable.

As a result of these experiments, I think it must be admitted that the negative is given to the statement that "In dealing with metals, whether uncombined or in salts, the order of their absorption for these rays is in fact that of their atomic weight, but the amount of absorption increases much more rapidly than the atomic weights themselves."

With the same weight of lithium and sodium the permeability is not far from equal, and for the same thickness of plate of metal sodium is not twice as absorptive as lithium, although its atomic weight is more than three times as great. Nor, in the case of comparing the same thicknesses, does sodium differ as much from lithium as potassium does from sodium.

My present experiments, taken along with the earlier ones, show that a line cannot be drawn between metals and non-metals as regards permeability. Chlorine is less permeable than sodium, but fluorine is more permeable. There seems to be a very rapid increase in absorptive power between the atomic weight of aluminium and that of potassium, and perhaps the same or a slightly less rate of increase on as far as manganese. Above manganese the variation is slight, and below aluminium not very great; but there seems to be the group of elements, carbon, nitrogen, oxygen, with perhaps fluorine and boron, that has a specially low absorptive power. Why carbon should be so much more like fluorine than the latter is like chlorine is a little difficult to say.

A phenomenon which I noticed before, but which I have since more fully investigated, without, however, arriving at what I consider an adequate explanation, is a peculiar granular structure, often exhibited by photographs of powders. In order to see whether the granulation was due to the powder being in lumps, instead of being reduced to an impalpable fineness, I experimented with three grades of powder:—coarse (passing through a ten-mesh sieve but not through a forty-mesh), medium (passing through a forty-mesh but not through a seventy-mesh), and fine (passing through the seventy-mesh sieve). The materials were mainly cryolite and magnesite, and eight different boxes of each were taken as below:—

1. Two grms. coarse powder.
2. Four grms. coarse powder.
3. Three grms. medium powder.
4. Three grms. fine powder.
5. $1\frac{1}{2}$ grms. coarse at bottom, and $1\frac{1}{2}$ grms. fine on top.
6. $1\frac{1}{2}$ grms. fine at bottom, and $1\frac{1}{2}$ grms. coarse on top.
7. $1\frac{1}{2}$ grms. fine and $1\frac{1}{2}$ grms. coarse, mixed.
8. Three grms. coarse, raised 1 c.m. from the photographic plate.

The granulation seemed to correspond with the size of particles, and was quite distinct even through the 4 grms., which made a layer not far from a centimetre thick. When the fine and coarse were mixed, the granulation of the coarse still showed; and when the coarse and fine were separate, the granulation was most distinct when the coarse was at the bottom. When the coarse powder was on top, the appearance in the photograph was very similar to that obtained from the mixed powders. When the box containing the coarse powder was raised a centimetre from the plate, the granulation was very much blurred. This is doubtless due to the overlapping of shadows, and this would account for the greater distinctness mentioned above, when the coarse powder was next the plate and the fine powder on top.

The phenomenon is certainly peculiar, because one would imagine that the particles would lie together in such a way that the average thickness would be the same. This is especially the case when the coarse powder is mixed with the fine, the latter (one would suppose) filling in the interspaces. It is true that a crystal of cryolite about 2.4 m.m. in thickness, placed in 2 grms. of cryolite fine powder, the total thickness being about 7 m.m., was outlined in the photograph, it being slightly less permeable than the powder that would occupy the same space.

Indeed, when the cryolite powder was mixed with about half its weight of calcite powder, the crystal was still fairly well outlined, though not so strongly marked as when cryolite powder alone was used; and I think a mixture could probably be made such that the powder, when pressed into a pretty compact mass, would just equal the crystal in absorbent power. When I had the mixed cryolite and calcite powder it showed a granular structure, though both minerals were so fine that they singly did not show granulation. This, doubtless, was due to an imperfect mixture, little lumps of one or other mineral being unbroken. I fancy that if the mixed powders had been sifted through a fine sieve, the granulation would not have shown.

There was in one box three pieces of cryolite crystal, crossed in such a way that one, two, and three thicknesses were exposed to the rays. The space not taken up by the crystalline pieces was filled in with cryolite powder.

In the photograph the three thicknesses were quite distinctly marked.

I suppose anyone would expect that if ordinary light were passed through a transparent substance, in the same way as was done with the Röntgen rays in the above experiments, no granulation would appear in the photograph. In order to make certain that such is the case, I made use of glass, in fine and coarse powder and in the form of beads of a somewhat larger size. Burning magnesium wire was used for illuminating.

When the photographic plate was developed, it was found, as was expected, that there was no granulation in any case. Of course, ordinary light is refracted and reflected by every particle of the glass, and perhaps we must consider that the different character of the Röntgen rays in this respect is the cause of the granulation shown on the photographic plate; perhaps the particles do not give an average thickness, and the interspaces are distributed irregularly.

A fact which would seem to corroborate this idea was that when a grm. of coarse calcite was mixed with a grm. of medium cryolite and fine magnesite, the dark spaces were larger in the photograph; the spots showing the less permeable calcite being farther apart than when coarse powder only was taken. It does seem strange, however, that the granulation should so nearly correspond to the size of the particles. This was seen most plainly in the case of halite and calcite, perhaps because of the less permeability of these minerals. The bottom layer may give the final impress, and where the particles are least permeable this impress is strongest. I cannot say that I am quite satisfied with this explanation, but no better has yet occurred to me.

The granulation shows so plainly that it could not escape the notice of any one who looks at the photographs in my possession, except where the powder was of impalpable fineness; and if observations made with a photometer did not reveal such a granular structure, it would merely show that the eye is more delicate than the photometer if the eye is assisted, as it was in my experiments, by the sensitive plate being kept covered by the lead sheet. I found this method vastly more sensitive than the one in which the substances formed the absorbent material, the rest of the photographic plate not being shielded from the Röntgen rays.

Kingston, Ontario.

THE ATOMIC MASS OF TUNGSTEN.*

By WILLETT LEFLEY HARDIN.

(Concluded from p. 157).

The next line of investigation was to make a number of determinations with material obtained from different minerals and different localities.

Reduction of Tungsten Trioxide obtained from Scheelite from New Zealand.

The oxide was extracted from this mineral and purified in a manner similar to that described under wolframite. No trace of molybdenum was found even before the oxide was heated in hydrochloric acid gas. The results of seven reductions are as follows:—

	Weight of WO ₃ . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	3'41018	2'70410	183'83
2	2'99000	2'37084	183'80
3	3'11613	2'47047	183'67
4	4'32830	3'43118	183'56
5	4'66735	3'70050	183'72
6	4'29620	3'40623	183'71
7	3'39104	2'68885	183'80
8	2'93215	2'32515	183'87

The mean of this series is 183'745. The maximum deviation is 0'20. Considering the number of experiments, this is the most concordant series of results ever obtained by reducing the trioxide of tungsten and weighing the resulting metal. In Experiments 2 and 7, the metal was cooled in a vacuum; in all the other experiments it was cooled in hydrogen.

Oxidation Series.

The metal obtained in the preceding reductions was used in these oxidations. The results of six experiments are as follows:—

	Weight of W. Grms.	Weight of WO ₃ . Grms.	Atomic mass of tungsten.
1	2'70219	3'40775	183'83
2	2'36771	2'98620	183'75
3	2'46705	3'11016	184'13
4	3'42163	4'31472	183'90
5	3'40086	4'28890	183'82
6	2'68249	3'38145	184'20

This series gives a mean of 183'94, with a maximum difference of 0'45.

Reduction of Tungsten Trioxide obtained from Wolframite from Connecticut.

The oxide was obtained from this mineral and purified by the method already described. The details of the work were the same as in similar series which precede. The results were as follows:—

	Weight of WO ₃ . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	3'14520	2'49330	183'58
2	3'10516	2'46141	183'51
3	4'17792	3'31244	183'83

Mean = 183'64

Maximum difference .. = 0'32

Oxidation Series.

The metal was obtained from the preceding reactions.

	Weight of W. Grms.	Weight of WO ₃ . Grms.	Atomic mass of tungsten.
1	2'48088	3'12790	184'05
2	2'44588	3'08318	184'22
3	3'29370	4'15260	184'06

The mean of these results is almost one-half a unit greater than the mean of the reduction series. It seems that the results from oxidations are invariably higher than those obtained by reduction.

Experiments on Material obtained from Hübnerite from Colorado.

The usual method of purification was used. Two reductions of the trioxide gave the following results:—

	Weight of WO ₃ . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	1'83600	1'45618	184'03
2	4'31878	3'42450	183'81

The metal resulting from these reductions was re-oxidised.

	Weight of W. Grms.	Weight of WO ₃ . Grms.	Atomic mass of tungsten.
1	1'45184	1'83090	183'85
2	3'40470	4'29225	184'14

Experiments on Material obtained from Scheelite from Bohemia.

The oxide was extracted and purified by the usual method. Two reductions were as follows:—

	Weight of WO ₃ . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	2'77363	2'19950	183'89
2	2'13327	1'69120	183'63

The re-oxidation gave:—

	Weight of W. Grms.	Weight of WO ₃ . Grms.	Atomic mass of tungsten.
1	2'18985	2'76060	184'17
2	1'68208	2'12070	184'08

Throughout this work, it had been noticed when tungsten trioxide was heated in a current of hydrochloric acid gas for some time that a considerable sublimate was formed, even when molybdic acid was absent. Enough of this sublimate for an atomic mass determination was obtained as follows:—Tungsten trioxide was heated for some time in a current of hydrochloric acid gas at a temperature of about 400°. The sublimate was removed from the tube, strongly ignited, and gently re-heated in a current of hydrochloric acid gas. The small white sublimate formed did not respond to the test for molybdic acid. The portion left in the porcelain boat was removed from the tube and strongly ignited in the air for a period of ten hours. It was then reduced in a current of hydrogen and the following result obtained:—

	Weight of WO ₃ . Grms.	Weight of W. Grms.	Atomic mass of tungsten.
1	1'12970	0'89610	184'13

Upon re-oxidation, this metal gave 184'87 for the atomic mass of tungsten.

The results from the sixty-four determinations made in the present investigation show a maximum deviation of one and a half units. A discussion of these results, with a view of arriving at the true atomic mass of tungsten, would be useless. To take the mean of all the results would be entirely unsatisfactory, and yet there seems to be no reason why any one result should be accepted in preference to any other. It will be noticed, in several instances, that three or four consecutive results agree very closely. These different series of concordant results, however, do not agree. The variations in these results are similar in every respect to those in the results of earlier experiments.

Various causes suggest themselves as possible factors in producing these variations. The lower values obtained in the latter part of the investigation are undoubtedly due to a better form of apparatus and a higher temperature.

The reductions were all made in a porcelain boat.

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xix., No. 8.

During each determination the boat increased in weight by from 1 to 3 m.grms. It is difficult to determine whether this absorption of tungsten by the boat would affect the results or not. If the tungsten is absorbed as metal it would produce no effect on the results, if not absorbed as metal it would. It was shown in the first part of this investigation that the metal obtained in the reductions contained silica. This may, in part, account for the higher values obtained in the oxidations. In view of these objections to the use of porcelain, a series of reductions were made in which a platinum boat was used. This, however, did not remove the difficulty; platinum absorbs tungsten and tungsten absorbs platinum, and the results obtained were just as variable as those obtained with the porcelain boat.

A series of observations on tungsten trioxide were next made with a view of determining whether or not this compound is suitable for atomic mass determinations.

The first point was to determine how rapidly this compound takes up moisture from the air. Several series of observations were made, and it was found in each case that the absorption of water was inappreciable. The rate at which the water was absorbed is best shown by the following series of weighings of tungsten trioxide, which had been left for several days in the open air. The oxide was first strongly ignited, then placed in a porcelain boat, carefully protected from dust, and left for four days in an open window.

Weight of the oxide at the beginning	5.34600 grms.
„ after one day.. ..	5.34605 „
„ „ two days	5.34620 „
„ „ three days	5.34625 „
„ „ four days	5.34630 „

From these observations it is evident that no appreciable error can be introduced by the absorption of moisture during the weighing of this compound.

A series of observations was also made to ascertain the action of light on this oxide. A weighed quantity of the material was placed in a desiccator and left for some time in direct sunlight. Weighings made at different intervals showed that no reduction had taken place. In working with this compound it is unnecessary to cover the desiccator with a black cloth.

Upon examining the porcelain tube after a reduction, a slight sublimate was usually noticed. Whether the tungsten trioxide is volatile at that temperature, or whether the moisture formed in the reduction carried mechanically small particles of the oxide from the boat, was not determined. In either case an error is introduced, but in all probability a very small one.

A series of observations was next made to determine whether or not tungsten trioxide contains nitrogen. A number of reductions were made in the usual way and the products set free were conducted through a U-tube containing pure water and a few drops of Nessler's reagent. The oxide used was obtained by strongly igniting ammonium tungstate for two days. Hydrogen was allowed to pass through the reduction apparatus for some time in order to completely remove the air. When the reduction was started, the solution in the U-tube began to assume a yellowish colour, even when the temperature was comparatively low. Before the reduction was half completed, the solution was of a deep yellowish brown colour. In some instances a slight precipitate was formed at the surface of the solution. Several observations were made, and the ammonium test distinctly obtained in each case. A series of blank experiments were made and no colouration was produced. The experiments proved conclusively that the oxide obtained by the ignition of ammonium tungstate contains nitrogen. No attempt was made to determine the quantity of nitrogen present. The oxide obtained by the ignition of metal was also examined and found to contain a trace of nitrogen. Whether the nitrogen in the former oxide was present as an oxynitride or as an ammonium residue, was not determined. If it

exists as an ammonium residue, then hydrogen must also be present. A number of experiments were made by fusing the oxide with lead oxide and also with anhydrous sodium carbonate, with a view of converting any hydrogen present into water. Nothing definite was established, but there were some indications that a small quantity of water was formed. If the nitrogen is present in large enough quantities to affect the atomic mass determinations, it would probably lower the results and also produce variations, for it is not likely that the quantity would be the same in all cases.

In regard to the occlusion of hydrogen by the metal, nothing definite was established. The results obtained by cooling the metal in a vacuum were practically the same as those obtained when the metal was cooled in hydrogen.

It has been shown in the foregoing observations that tungsten attacks the vessels in which the atomic mass determinations have been made, that the oxidation of tungsten is either slightly volatile, or that a small portion is carried mechanically by the water formed in the reductions, and that the supposed trioxide of tungsten contains nitrogen and probably hydrogen. In view of these facts and of the fact that there is no means of determining when the reduction of oxide to metal is complete, and finally, in view of the fact that more than one hundred and fifty determinations have been made of this oxide, and nothing definite established, it is evident that the atomic mass usually employed in the determination of the atomic mass of tungsten must be regarded as unsatisfactory.

SEPARATIONS WITH ALKALINE ACETATES.

By HARRY BREARLEY.

(Continued from p. 51).

III.—Cobalt and Manganese from Iron.

IN view of the large number of tests it is desirable to make on the separation of each element under various conditions, it is important to choose the most rapid—if at the same time undoubtedly accurate—means available of estimating that element.

With the view of making subsequent tests more intelligible, the method of estimating the separated cobalt is here set forth.

It has been previously noticed that in estimating nickel by means of potassium cyanide and silver iodide, if cobalt be present it will be estimated along with the nickel; but, so far as I can find, nobody has determined whether the titration which serves so accurately for nickel is equally applicable to cobalt. The suggestion, however, is so forcible that it must have been attempted by most people who have made the nickel titration.

In attempting the titration of cobalt solutions, under the conditions previously used, the first noticeable change is the immediate darkening of the solution on adding the cyanide. If the cyanide be further added without unnecessary delay it is seen that the AgI indicator becomes slowly dissolved, and that some time before enough cyanide has been added to combine with all the cobalt. On allowing the just-cleared solution to stand, the AgI turbidity reappears; a few more drops of KCN clear it, further standing causes it to reappear, and so on, but not indefinitely. A point is quickly reached at which the solution can stand for hours without becoming turbid; or if an excess be added in the first instance, there is no turbidity on standing. In this latter way—that is, by adding cyanide in excess, allowing to stand awhile, and going back with silver nitrate—it is easy to obtain consistent values when titrating similar amounts of cobalt.

The darkening of the solution just noticed is not permanent. The usual change is from the deep golden brown to a salmon-coloured liquid which very gradually

bleaches. The colour changes, however, are not always the same, except in identical liquids treated alike. The precise colour effect of each reagent has not been determined, but the addition of potassium iodide before or after the cyanide gives distinctly different tints.

It is practicable to replace ammonium by the corresponding sodium salts, and to make the final alkalinity with sodium carbonate. This change gives us a colouration which is neither so deep nor so variable, and the disappearance of the precipitated carbonate on adding cyanide is some indication of the progress of the reaction. Personally, this soda modification has proved eminently satisfactory, almost rivalling in delicacy the cyanide titration of nickel; in other hands it has not behaved so well. A comparison of soda and ammonia salts, kindly made by a friend, led him to prefer the latter.

Unfortunately whether soda or ammonia is used, the dilution of the solution titrated is not without influence. Results from two sets of solutions are appended.

TABLE VIII.

	Volume of solution. C.c.			
	120.	200.	300.	400.
Ammonia..	17.84	17.52	16.92	— c.c. KCN
Soda ..	18.22	17.93	17.58	17.14 " "

The tests in each set varied only in volume. They contained the same total quantity of free alkali, so that the proportion of this reagent is less in the larger volumes. This difference would tend to give more concordant values.

Lacking at present the recommendation of extended use in various hands, it was deemed undesirable to use this method alone and unchecked. Corresponding titrations were therefore made here and there according to Winkler's method (see "Fresenius's Quant. Anal.," or "Sutton's Vol. Anal."), which is regarded as "thoroughly satisfactory for technical purposes" at least. It may be worth while to notice that, contrary to expectations, there was no noticeable trouble experienced on account of the organic (acetic) acid.

Any error due to traces of manganese in the bar iron was exactly balanced by treating a sample—lacking cobalt—exactly like the test, and standardising the permanganate or cyanide in an equal portion of the filtrate.

The details of the separation to be now applied to cobalt are the same as those followed with nickel and iron, and presuming an acquaintance with the Ni titration, that for cobalt may be summed briefly:—The idea is to get an excess of cyanide into the solution, but not a large excess. Make the solution alkaline, and then, without unnecessary delay, add cyanide to completion or thereabouts; add the indicator ($KI + AgNO_3 + Am_2SO_4$ or Na_2SO_4), and then, if necessary, enough cyanide to clear the solution and 2 or 3 c.c. in excess. I prefer to use a very dilute solution of silver nitrate—so dilute that 10 c.c. is only equal to 1 c.c. KCN where about 16 KCN equals 0.01 gm. cobalt. The KCN is standardised with a similar amount of cobalt in exactly the same way, and the two are allowed to stand awhile—say, fifteen minutes. If they are then perfectly clear,* silver nitrate is added until the turbidity reappears. This is most satisfactorily accomplished by allowing the nitrate to run down the side of the flask. A very copious turbidity is soon formed on the surface of the solution, and the readiness with which this disappears on shaking is some indication of the amount of silver nitrate yet to be added. It is easier, too, in this way to note the first appearance of the turbidity. 2 c.c. of the silver nitrate makes a decided difference in half a litre of solution; 2 c.c. $AgNO_3$ equals 0.2 c.c. KCN, equals 0.00013 gm. cobalt.

Samples containing 1 gm. (separations will always be

* A slight turbidity, due to traces of manganese, should be filtered off if it is so decided as to prevent the AgI turbidity being readily recognised.

from 1 gm. unless otherwise stated) of iron and 0.1 gm. of cobalt gave the values shown in—

TABLE IX.

	Method of titration.	
	KCN.	Winkler.
Ammonia salts	0.0995	—
Soda salts	0.0997	0.1015

Nor is the separation less perfect with other proportions of cobalt. In parallel columns there are arranged some results by Messrs. Leffler and Jervis, to whom my best thanks are due. Apart from their confirmatory value, such results are testimony to the fact that the method may be satisfactorily worked without lengthy experience. Their results are with ammonia salts throughout, and on mixtures of bar iron and cobalt nitrate. My own results are with soda salts throughout.

TABLE X.

Added. Gm.	Cobalt found.		
	Brearley.	Leffler.	Jervis.
0.0200	—	0.0196	0.0198
0.0300	—	0.0294	0.0301
0.0500	—	0.0496	0.0508
0.1000	0.0997	0.1001	0.0995
0.2000	0.2006	—	—
0.3000	0.2982*	—	—

* Titration irregular.

For the rest, it remains only to notice a few variations similar to those observed with the preceding metal. To prevent repetition, the corresponding tables in the nickel series will be placed in brackets so that the details of the tests can be referred to.

Further addition of acetate may be made to the heated solution (p. 50, col. 1).

TABLE XI.

	Co added.	Found.
Soda salts	0.1000	0.1003
Ammonia salts	0.1000	0.0997

Prolonged boiling introduces no error (VI.).

TABLE XII.

Boiled.	Cobalt added.	Cobalt found.
15 minutes	0.1000	0.0997
30 "	0.1000	0.1000

Deservedly or otherwise, the separation of cobalt from iron by means of acetates is less favourably looked upon than the corresponding separation of nickel. Thus, T. Moore (CHEMICAL NEWS, lxx., 75), who seems to have made a special study of these two elements, says:—"A very short experience will suffice to demonstrate that the basic acetates of iron must be re-precipitated at least four times before one can be assured that the separation, so far as cobalt is concerned, is complete."

Whether cobalt is less readily separated from iron than is nickel would be seen by adding varying amounts of acetate in excess and, making every other condition alike, noticing in which the percentage recovery fell the more rapidly. The results of such experiments are arranged in Table XIII. The agreement between the temperatures of turbidity should not be overlooked. It afforded additional evidence that the two sets of solutions were very similar. The minimum amount of acetate for solutions made up as these were would be about 12 c.c.

TABLE XIII.

Acetate. C.c.	Percentage recovery of—		Respective Temp. turbidities.
	Nickel.	Cobalt.	
20	99.0	99.04	72°, 70° C.
50	95.2	97.5	60°, 59° C.
100	90.0	93.8	53°, 54° C.

This table can claim to answer the question at issue only for the particular *modus operandi* used, which was that generally adopted for the previous separations. It does not necessarily follow that the same order would hold whatever variations might be introduced, though for two so closely related elements as nickel and cobalt the result may perhaps be considered generally true.

A table similar to XIII. will be given later embracing all the metals whose separations from iron have been attempted.

Separation of Manganese.

A previous paper on the estimation of manganese in *spiegels* (CHEMICAL NEWS, lxxv., 13) makes it needless to repeat any array of experiments. This circumstance affords us an opportunity of looking about more casually than it might otherwise be thought proper to do. The paper mentioned contains an error of some moment; a few words will set things right. The use of ammonium acetate is restricted, because in heated solutions salts of ammonia react with the permanganate in the subsequent titration. It may be noticed that as much as 10 c.c. of strong ammonium acetate is used to precipitate the one grm. of iron; this is several times larger in amount than the soda acetate used, or than it need be. This correction made, the danger of the permanganate reaction would be lessened.

It is noteworthy that while the separation of nickel and cobalt from iron by means of acetate are commonly deprecated, and are fast disappearing from the arena of usefulness, the separation of manganese by these means has, on the whole, and particularly for low percentages, been well spoken of. This argues in a general way that the former metals are less accurately separated from iron under similar conditions than is manganese. Indeed, if a series were arranged parallel with those first given (Table XIII.), it would be seen that as perfect a separation accompanied the larger amount of acetate as the smaller. In fact, Mr. Jervis has, in this laboratory, increased the acetate to 200 c.c., and finds still a perfect separation. The instructions, therefore, for separating iron and manganese perfectly and without re-precipitation, may be condensed into a word—ditto.

One frequently hears the use of soda salts in the gravimetric estimation of manganese justified by the belief that they give a better separation than the corresponding ammonium salts. There is no doubt but that either will give a perfect separation when used sparingly enough. That there could be any question of their comparative merits reminds one of the careless way in which the acetates are generally used—as though an excess were of no moment, or perhaps improved the separation.*

The relative efficiency of these two acetates was tested by using such large excesses as to cause imperfect separations. As the point is not an important one, it may be sufficient to notice that the result was so slightly in favour of ammonium acetate that they may be regarded as practically equal. It is to be feared that the higher results with soda acetate are sometimes due to the well-known difficulty of washing away the non-volatile precipitant.

It appears plainly that alkaline acetates have been ill-spoken of and neglected, not because of any inherent shortcoming in the reagents themselves, but on account of the thoughtless way in which they have been used. There are other highly-prized reagents which would fail under like treatment. That I may not be accused of unduly magnifying this error of excessive acetate, I quote below a few instances from such recently described methods as retain this means of separation. The figures in brackets give the approximate volume if diluted to such strength as is used throughout this investigation.

* Parry and Morgan (*Industries*, 1893; also CHEMICAL NEWS, lxxv., 308), respecting the separation of nickel and cobalt from iron, recommend that the solution be "made neutral with soda carbonate and the iron precipitated by the addition of a large excess (italics copied) of soda acetate and the solution boiled for some time" (italics not copied).

Parry and Morgan (CHEM. NEWS, lxxv., 295, 1893)—250 c.c. hot ammonium acetate, strength not stated.

Arnold ("Steel Works Analysis," 1895)—Half a grm. of *spiegel* is precipitated with 20 c.c. [260] ammonium acetate. And 25 c.c. [more than 250 c.c.] saturated solution of sodium acetate is used to separate 0.1 grm. nickel from similar quantity of iron.

Dittmar ("Quant. Chem. Anal.," 1887)—"Add acetate of ammonia solution (not too little)."

Phillips ("Engineering Chemistry," 1891)—later edition not available—Half grm. of *spiegel* precipitated with 20 c.c. [290] 5 E ammonium acetate.

Hiorns ("Practical Assaying," 1892)—1.3 grms. steel 20 c.c. [260] acetate.

These large excesses become more curious when it is noticed that (with the exception of Phillips) strong emphasis is usually laid on the necessity of neutralising with great exactness.

The last writer (Hiorns) says:—"If the basic acetate precipitate be dark red an insufficient amount of acetate has been added, and more of that salt must be introduced" (p. 397). I have previously heard this remark made to beginners in laboratory practice, though it is rarely repeated in text-books. Altogether apart from the peculiar appearance due to iron in the ferrous state, the basic acetate may vary from a fine compact brick-red precipitate, which remains long suspended in the solution and can be eliminated only by repeated filtration through ordinary paper, to an open flocky purple-brown precipitate, which settles readily and leaves a crystal clear solution. The former occurs in presence of large volumes of acetic acid, the latter by precipitating with little more than minimum acetate. Similar precipitates, and any variety between, can be produced with other amounts of acetate. The inference is that the colour of the precipitate alone is of no value in determining whether enough acetate has been added or not.

(To be continued).

NOTES ON THE ESTIMATION OF SILVER IN SILVER-PLATING SOLUTIONS.

By T. J. BAKER.

I. THE method of weighing the precipitate obtained with HCl is untrustworthy in the case of old solutions, unless precautions are taken to separate the copper cyanide and other impurities precipitated along with the silver chloride.

II. THE method of fusing the impure silver chloride with sodium carbonate and nitre is often attended with the disadvantage of small globules of silver adhering to the crucible.

III. THE following method devised by the writer avoids the disadvantages referred to above:—

Precipitate say 50 c.c. of the solution by boiling with slight excess of nitric acid. The precipitate will contain all the silver as silver cyanide, together with copper cyanide and other impurities. Wash and dry the precipitate.

Burn the filter-paper, and wrap up the ash, together with the precipitate, in assay lead. Compress into a small bulk, and cupel, employing the usual precautions of duplicate assays and checks.

Note.—Silver cyanide is completely reducible to metallic silver by simple ignition, and the effectiveness of cupelling the precipitate depends upon this reaction.

The method has been attested in the metallurgical laboratories of the School, and has given uniformly good results.

Electro-Metallurgical Laboratory,
Birmingham Municipal Technical School,
September 2, 1897.

ON THE
PERMEATION OF HOT PLATINUM BY
GASES.

By WYATT W. RANDALL.

IN connection with a research which has been in progress for some time, and in which the attempt is being made to prepare absolutely pure hydrogen for spectroscopic examination, the author has endeavoured to free the gas from impurities by filtering it through a diaphragm of hot platinum. The spectroscopic results obtained will be published later; it seemed worth while, however, to give at this time a short account of the work which has been done in the past in the matter of the permeation of metals by hydrogen, and also of the measure of success which has attended the attempt to free the gas from impurities by the method mentioned.

The fact of the permeability of hot platinum by hydrogen was first announced by Deville and Troost (*Compt. Rend.*, lvi., 977, 1863; *Phil. Mag.* [4], xxvi., 336; *Abstr. Jsb.*, 1863, 23; *CHEM. NEWS*, vii., 294). When hydrogen was introduced into a hot platinum tube surrounded by one of porcelain filled with air, the former gas passed through the walls of the metal tube, but no passage of air into the inner tube could be detected. Later papers by the same authors (*a*, *Compt. Rend.*, lvii., 894, 1863; *b*, *Ibid.*, lvii., 965; also *Ann. Chem.*, Liebig, cxxx., 254; *c*, *Compt. Rend.*, lix., 102, 1864; *Abstr. Jsb.*, 1864, 89; *Phil. Mag.* [4], xxviii., 229; *CHEM. NEWS*, x., 57) established the results claimed, and showed that iron also was permeable by certain gases at high temperatures.

A year or two later Graham (*Phil. Trans.*, clvi., 399, 1866; *Phil. Mag.* [4], xxxii., 503; *J. Chem. Soc.*, xx., 257; *Abstr. Proc. Roy. Soc.*, xv., 223) repeated the experiments of Deville and Troost, taking, however, greater precautions to avoid error. His method differed in that, by means of a Sprengel pump, a vacuum was maintained within a platinum tube, which was closed at one end, and which was heated in an atmosphere of hydrogen. The gas was found to penetrate rapidly into the interior of the tube. When air was substituted for hydrogen, practically no gas was delivered by the pump, but Graham was of the opinion that air did penetrate to a slight extent. As, however, the connections of his apparatus were not above suspicion, and as he employed air which had been dried by sulphuric acid alone, the evidence for the penetration by air is by no means convincing. At the temperature employed, any water-vapour present in the air would probably have been at least partially dissociated and free hydrogen have been formed. The same objection might hold good for all his experiments in this series. When carbon dioxide was tried 0.3 c.c. of gas was collected by the pump, and "an indeterminate small portion of this was condensed by baryta water and appeared to be carbonic acid." In view of the power of carbon dioxide to cling to the walls of a vacuous glass tube, and to be but slowly removed, the evidence here, again, is inconclusive. Negative results were obtained with chlorine, hydrochloric acid gas, steam, and ammonia. When coal-gas containing hydrogen, methane, carbon monoxide, and ethylene was employed, hydrogen penetrated, but no evidence of carbon compounds could be obtained on exploding the gas collected with oxygen. Graham concluded that a little nitrogen might penetrate the platinum wall, provided hydrogen was passing through at the same time in the opposite direction, since he obtained evidence of such penetration when the tube was filled with hydrogen and heated in an atmosphere of nitrogen.

In accordance with conclusions arrived at by a study of the passage of gases through various septa, Graham assumed that the penetration of platinum by hydrogen was due to the liquefaction of the gas on the surface, the diffusion of the condensed gas through the material of the wall, and the evaporation of the liquid from the inner surface of the tube into the vacuum.

Nearly all research in this field since the publication of these results has had to do with the nature of the occluded gases, and not with questions of penetration considered by itself. Morse and Burton, however, have shown* that the hydrogen contained in the flame of an ordinary blast-lamp will penetrate a platinum vessel heated by that means; and Ramsay, in studying the phenomena connected with the passage of hydrogen through a palladium septum, has found (*Phil. Mag.* [5], xxxviii., 206, 1894) that this power was apparently possessed by no other gas examined. As palladium is permeable to a far higher degree than platinum, it would naturally be assumed that the latter metal would resist the passage of all gases except hydrogen completely.

In his studies of the properties possessed by hydrogen when occluded in platinum and palladium, Graham found that the metals could be more highly charged with the gas by making them serve as the negative pole of galvanic cells, than by heating them and allowing them to cool in an atmosphere of hydrogen (*Proc. Roy. Soc.*, xvi., 422, 1868). "The occluded hydrogen," he says, "is certainly no longer a gas, whatever may be thought of its physical condition." Oxygen was not occluded when the current was reversed. The evidence, he considered, was on the whole against the view that chemical combination takes place between the metal and the gas. Careful examination of the change in volume suffered by palladium and its alloys on absorbing hydrogen led to the view that the gas was converted into a metal-like substance and formed a species of alloy with the palladium (*Proc. Roy. Soc.*, xvii., 212, 500, 1869; *Compt. Rend.*, lxviii., 1511). Hydrogen in this particular condition—"Hydrogenium," as Graham calls it—is magnetic, shows electric conductivity, and possesses some tenacity. Its density varies: in palladium alloys it is 0.711—0.7545; in palladium itself, 0.854—0.872.

This conclusion was, however, challenged in the more recent work of Berthelot (*Compt. Rend.*, xciv., 1377, 1882; *Ann. Chim. Phys.* [5], xxx, 519), who, as a result of thermo-chemical investigations, returned to the view that definite complex hydrides of palladium and platinum are formed. The results obtained by Thoma (*Ztschr. Phys. Chem.*, iii., 69, 1889) had to do with the absorption of the gas considered as a case of solution capable of supersaturation. The conclusions of Berthelot have been offset by a research by Mond, Ramsay, and Shields (*Phil. Trans.*, 186 A, 657, 1895; *Abstr. Proc. Roy. Soc.*, lviii., 242) upon platinum-black, in which doubt was cast upon the purity of the material used by the French chemist, and evidence given to show the untrustworthiness of his thermo-chemical data for establishing the condition of the occluded gas.

The method of spectroscopic examination seemed capable of yielding so much more accurate results in determining the presence of foreign gases in the filtered hydrogen than any of the analytical methods to my knowledge used hitherto in this work, that it seemed worth while to put it to trial. Tubes filled with gas were examined in the Physical Laboratory of this University, with the aid of a large concave grating specially prepared for such work, and photographs were made of each in turn. For their kindness in making these examinations I am indebted to Dr. J. S. Ames and Mr. W. J. Humphreys.

The essential features of the apparatus used in the production of the gas to be tested are represented in the accompanying figure. It consists of a platinum tube, A A', about 350 m.m. long, and of about 3 m.m. internal diameter, closed at one end and sealed at the other, by means of fusible glass, into a soft-glass tube, which is in turn connected with the sparking-tube B and the Töpler pump. The closed end of the platinum tube projects into a piece of hard-glass tubing, B B', through which a current

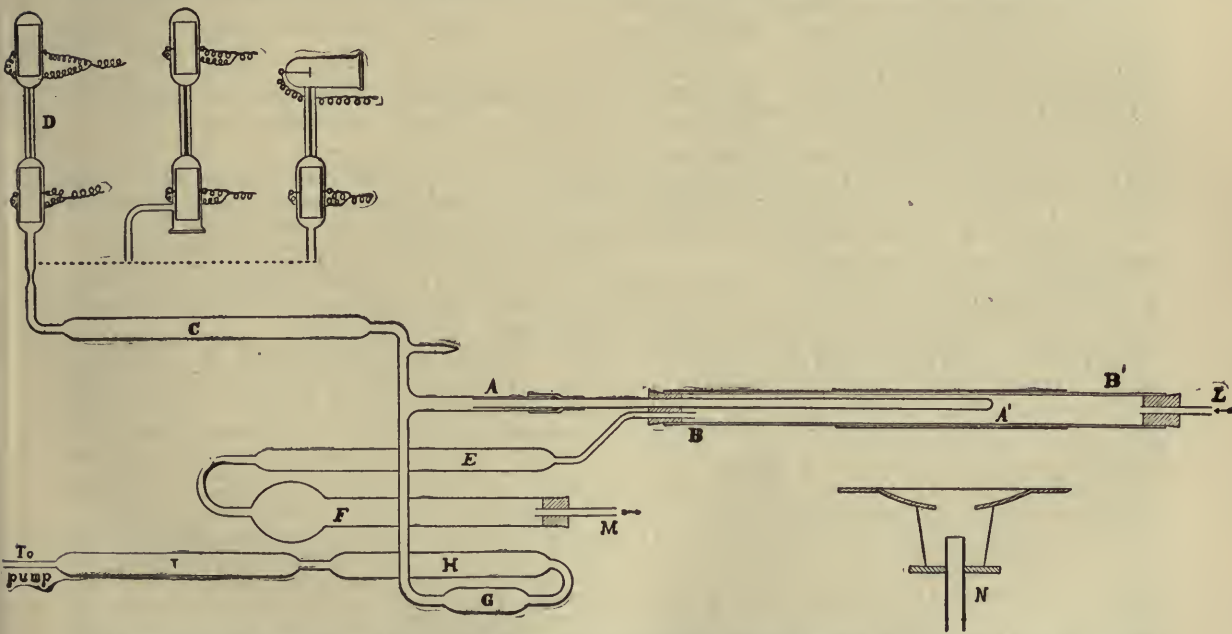
* *American Chemical Journal*, x., 148 (1888). It is to Professor Morse that I owe the suggestion of employing a platinum septum in this work.—W. W. R.

of dry gas, entering at L, could be passed, while at the same time the platinum tube can be heated by means of a Bunsen burner, N, below the combustion-tube. The latter is covered for a portion of its length with a layer of thick copper foil to distribute the heat, and, with the aid of asbestos-board screens, the temperature of the two tubes can be raised to a white heat. A plug of asbestos was pushed into the annular space between the two tubes to give support. A short piece of black rubber is drawn over the place where the platinum tube is sealed into the glass tube, is wired on and well shellaced, to guard against possible leakage from fracture.

The tube c is filled with phosphorus pentoxide. The gas entering at L has been made to pass through sulphuric acid, calcium chloride, soda-lime, and, finally, through a long tube filled with phosphorus pentoxide. It passes out at B through the guard-tubes E containing the pentoxide, and F containing calcium chloride and soda-lime. When hydrogen was the gas employed, it was first passed through four wash-bottles filled with acid, and two filled with alkaline permanganate solution, and then through a tube filled with red-hot copper turnings, on its way to the drying-tubes. It was hoped the latter device would free the gas from any admixture of oxygen, whether present as a constituent of air, or given off from the permanganate solution (see footnote at end of paper).

of course taken to have the air as dry as possible, in order to avoid the presence of hydrogen through the dissociation of water-vapour. The vacuum within the metal tube could be maintained apparently indefinitely under these conditions.

The tubes were filled with hydrogen as follows:—The carefully cleansed tube was sealed to the apparatus, and, when the air had been completely exhausted, was left for twenty-four hours or more; by this time all moisture was probably removed by the phosphorus pentoxide. By continuous sparking and exhaustion the gas occluded by the electrodes was then removed, and a condition of brilliant green fluorescence readily obtained. On raising the mercury in the Töpler pump so as to compress all the gas in its bulb and tubes into a space of not more than a fraction of a cubic centimetre, and at the same time noting the change, if any, which took place in the height of the mercury column in the delivery-tube,—in other words, on using the pump itself as a McLeod gauge,—it could be shown that the pressure within the exhausted space was not more than one or two millionths of an atmosphere. Connection between the pump and the rest of the apparatus was then cut off by turning a glass tap, and hydrogen was admitted slowly into the sparking-tube by heating the metal tube while a current of dry purified gas was circulating about it. When the pressure inside the



The sparking-tubes were of several forms, at first with wire electrodes, afterwards with cylindrical electrodes of aluminium tubing or foil. In some cases photographs were made of the spectrum of the light emitted through the walls of the capillary tube; in others, of that observed "end on" through a disk of quartz sealed as a window to one of the bulbs. In one case the tube was specially constructed so that the light observed through the window was that produced at the electrode and not that in the capillary tube; the light thus obtained was, however, so weak that no photograph could be made of the spectrum.

Having demonstrated the tightness of the apparatus, and the fact that at the temperature employed hydrogen is capable of permeating the walls of the platinum tube, experiments were made which afforded evidence that a similar power of penetration is not possessed by oxygen or nitrogen. Air was drawn through the apparatus while the metal tube was maintained at a white heat. Care was

sparking-tube had reached about 1 c.m., the heating was discontinued and the apparatus exhausted as before. The process of filling and exhausting was repeated from three to six times, after which the tube was filled to the desired pressure—which was between 3 and 8 m.m.—and was sealed off. In some cases, as an extra precaution, the tube was enclosed in a copper air-bath, and heated during the process of exhaustion to a temperature of 150° to 200°, the sparking being meantime continued.

It may be as well to say that the passage of the hydrogen into the exhausted space was very slow as compared with what the statements of Graham had led me to expect. It may be that the tube employed by me was thicker and more thoroughly hammered, and that the temperature obtained was not so high. At all events the pressure in the sparking-tube rose only at the rate of about a millimetre in three to five minutes, the volume of all the tubes and connections open to the gas being about 100 c.c. On this account the time required to cleanse,

exhaust, and fill the tube in the manner described, waiting in each case for diffusion to equalise the pressure throughout the long system of tubes, rendered the work very slow and tedious, especially as only a short time was available on any one day to be devoted to the work.

The sparking-tubes had at first, except for the tube c, been in direct connection with the Töpler pump and mercury gauges. On photographing the spectrum it was found that the gas was contaminated with the vapour of mercury. To avoid this difficulty the tubes G, H, and I were introduced between the pump and gauges and the system to be exhausted. I is a long tube filled with powdered sulphur, H a similar one containing bright copper turnings, and G contains gold-leaf. H and I are shown in the figure much shorter in proportion than they are in reality. So perfectly do these guard-tubes serve their purpose, that after one year the gold-leaf shows no signs of amalgamation. The copper has apparently held back all sulphur-vapours from the sparking-tubes, whereas the mercury in the manometers have become fouled from not being protected in its turn by copper. No trace of mercury-vapour has been detected in the gas contained in any of the sparking-tubes since the precautions mentioned were taken to exclude it.

Results.

A careful examination of the photographic negatives obtained, when the hydrogen produced as described and contained in the sparking-tubes was used as the source of light, has failed to reveal the presence of any other substance—unless the so-called "compound" spectrum is to be considered an indication of such contamination. The purified hydrogen drawn through the combustion-tube B B' probably frequently contained nitrogen, but no signs of that gas could be detected in the spectrum of what had passed into the inner tube. If the compound spectrum is due to the presence of water-vapour, it is not clear how it can be eliminated, no more efficacious drying agent than phosphorus pentoxide being available.

It may be well to mention here that the results obtained in the examination of the spectrum of hydrogen purified as described in this paper, do not seem to agree with those obtained by Messrs. Trowbridge and Richards (*Am. Journ. Sci.* [4], iii., 118; *Phil. Mag.* [5], xliii., 137, 1897). According to these authors, an oscillatory discharge from their powerful storage-battery through hydrogen did not yield the compound spectrum, whereas the direct discharge produced the compound spectrum as usual. In the experiments performed with hydrogen, as described in this paper, the discharge was that of a large Ruhmkorff coil, sometimes with and sometimes without a Leyden jar in the secondary circuit and with considerable variations of current in the primary coil. In no case was the compound spectrum absent. Messrs. Trowbridge and Richards do not mention the character of the tubes they employed, nor give any account of the method of purification used for the gas, or that of cleansing and filling the tubes. It is just possible that were these data available, some explanation of the difference in our results might be forthcoming. It is true that the pressures used in this work were on the average a little higher than those employed by Trowbridge and Richards, but not to an extent, in all probability, sufficient to account for so marked a discrepancy.

As has been stated, oxygen and nitrogen do not penetrate hot platinum under the conditions described above; it remained an interesting question to determine whether this was due in any degree to the relatively high specific gravity of these gases as compared with hydrogen. Accordingly an experiment was made to see if marsh-gas, with a density only half that of oxygen, would penetrate into the inner tube. A large gasometer was filled with the gas prepared by heating a mixture of dehydrated sodium acetate and soda-lime, and washed with water and acid sodium sulphite solution to remove the vapours of acetone. After standing several weeks in contact with

water in the gasometer, it was passed again through a solution of the sulphite, through soda-lime, calcium chloride, and sulphuric acid, to mix with the purified hydrogen as it entered the tube filled with hot copper turnings mentioned above.* After the air had been removed, and a gas mixture, consisting of about four-fifths marsh-gas and one-fifth hydrogen, was being passed through the combustion-tube B B', the latter was heated. Gas passed very slowly into the sparking-tube; after about three hours the pressure had risen to only about 3 m.m. Examination of the gas by means of a large prism spectroscope failed to show the presence of any carbon compound; but, on account of the low pressure the light was too feeble to give any result with the photographic method, even after a very long exposure, and so this experiment will still require further and more accurate confirmation.

It is proposed to continue this work with hydrogen and other gases.

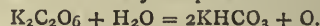
Since this paper was written a note has been published by Ramsay and Travers (*CHEMICAL NEWS*, lxxv., 253), in which they describe their attempts to pass helium and argon through septa of platinum, palladium, and iron, under conditions similar to those described in this paper. No evidence of such penetration was, however, obtained. —*American Chemical Journal*, xix., No. 8.

ON A

NEW CLASS OF OXIDISING SUBSTANCES: THE PERCARBONATES.

In a recent number of the *Zeitschrift für Electrochimie* MM. Constam and A. von Haussen announced the discovery of a new series of compounds which are of very great interest. We know that on electrolysis the alkaline carbonates, M_2CO_3 , we obtain hydrogen and the hydrate of the constituent base at the cathode, and at the anode oxygen and carbonic acid, which re-combines with a part of the base to form bicarbonate.

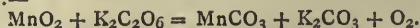
The authors have observed that if we electrolyse a saturated solution of carbonate of potash, and gradually lower the temperature, the disengagement of oxygen gradually diminishes at the anode, and finally ceases completely at about $-10^\circ C$. And further, instead of a crystalline bicarbonate being formed, we have a bluish amorphous powder, shown by analysis to consist of $K_2C_2O_6$; this is percarbonate of potassium. We can thus explain its formation: the carbonate of potassium in saturated solution first of all becomes dissociated into ions K and KCO_3 ; when electrolysis intervenes, the two ions KCO_3 unite, to form the body $K_2C_2O_6$. The phenomenon does not occur in dilute solutions, as the carbonate of potassium splits up into the ions K_2 and CO_3 . The percarbonate obtained in the above manner should be quickly thrown on a filter, and dried over phosphoric anhydride. It is very hygroscopic, and decomposes water at the ordinary temperature:—



When gently heated it decomposes according to the following equation:—



In the presence of oxidisable matters it acts as an oxidising agent. But it can also act as a reducing agent:—



* It is not generally known that comparatively large quantities of oxygen are set free when hydrogen is made to bubble through potassium permanganate solution. Unless care is taken to remove this oxygen, say with red-hot copper and a drying agent, an opportunity is given for a very serious error in all experiments where pure hydrogen is desired. This reduction of permanganate by hydrogen is now under investigation in this laboratory by Prof. Morse.—W. W. R.

From these reactions the authors conclude that this new body is in reality the neutral carbonate of a higher oxide, peroxide of potassium. Besides it produces, like the higher alkaline oxides and the alkaline earths, peroxide of hydrogen in the presence of acids. — *Revue Générale des Sciences*, No. 17, 1897.

CORRESPONDENCE.

SUPPOSED NEW ELEMENT WITH IRON.

To the Editor of the Chemical News.

SIR,—With reference to the article by Mr. G. G. Boucher (*CHEMICAL NEWS*, vol. lxxvi., p. 99), on the discovery of a possible new element with iron, I should like to point out that the reactions given by him in that article for his unknown metal are almost identical with those of molybdenum, the only exception being with Na_2SO_3 , which with dilute solutions gives no perceptible change as in his case, but with a fairly strong solution gives a blue colouration on boiling, while with SnCl_2 , in the case of the metal separated from iron, no change takes place; with the metal produced from boiler dust, SnCl_2 gives the usual molybdenum colourations of dark blue in the cold and brown on boiling with HCl ; and further, the most characteristic reaction for his unknown metal, viz., the intense blue colour produced by evaporation with sulphuric acid, is also one of the more characteristic tests for molybdenum, and therefore—seeing that from Mr. Boucher's description of the process for separating his unknown metal no steps are taken to remove molybdenum should such be present—I would suggest that this metal may prove to be molybdenum.—I am, &c.,

CHARLES H. JONES.

Laboratory, Minas de Rio Tinto, S. Spain,
September 3, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 10, September 6, 1897.

The Minister of Public Instruction and the Fine Arts addressed to the Academy the amplification of the decree by which the President of the Republic authorises the Academy to receive the donations offered by Henry Wilde for the foundation of an annual perpetual prize of 4000 frs. to be awarded to the person whose discoveries or researches in astronomy, physics, chemistry, mineralogy, geology, or experimental mechanics shall be judged most worthy of reward.

The Magnetic Deflection of the Cathodic Rays and of the X Rays.—G. de Metz.

Action of the X Rays on the Luminescence of Gases.—A. de Hauptinne.—A tube containing a gas at a low pressure becomes luminous under the action of electric vibrations, and becomes luminous at a much higher pressure if submitted to the action of the X rays.

Revue Générale des Sciences Pures et Appliquées.
No. 15, August 15, 1897.

This number contains no original matter of chemical interest.

Bulletin de la Société d'Encouragement pour l'Industrie Nationale. Series 5, Vol. ii., No. 7, July, 1897.

This number contains an account of the annual general meeting of the Society, which took place on June 25th under the presidency of M. Mascart, who delivered an address. The reports of various committees were presented and a number of medals conferred.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1976.

THE GOVERNMENT LABORATORY.

By CHARLES J. MEADS.

THE new Government Laboratories, which have been upwards of two years in construction, were formally opened on October 1st. They are situated in close proximity to King's College Hospital and to the rear of the New Bankruptcy Court, and occupy a site of 7900 square feet.

Outwardly the building is a plain one, of red brick; the rooms (which occupy three floors and a basement) are lofty and well lighted, their walls of faced bricks are white with a dado of pale blue; the floorings are of cement overlaid with pine blocks. Ventilation is effected by a fan in the basement, the means of artificially heating and lighting the establishment being steam and electricity respectively. There are three distinct water systems,—one in connection with the Water Company's main, a second supplied by a reservoir situated under the basement, and a third of cooled water, a small reserve of which is kept in a tank near the roof. The pipes conveying these services, as well as those of gas and steam, are easily accessible and naked throughout their length, and painted in different colours according to the use to which they are put. Those of each bench are separately connected with their several mains, so that, should any leakage occur in them, they can be thrown out of use without other than local inconvenience. Dirty waste liquid is conveyed away by V-shaped wooden troughs covered with lead thickly coated with paint; the difficulty of corrosion accompanying the use of leaden pipes for this purpose is thus avoided.

The basement is mainly devoted to store- and engine-rooms. A sample receiving room contains an ingenious bottle-washing apparatus. The steam leads of the boiler are fitted with reducing-valves for the steam drying and heating contrivances, the main work of the engine being the pumping of the water supplies, and driving the ventilating fan and also a carbon dioxide freezing machine, which provides the supplies of ice and cooled water. The reagents room, the mechanical laboratory, the water analysis room, and the hydrometer room, in which all the instruments in use are standardised, also partly occupy the basement.

The ground floor is allotted to the rooms of the Principal and Deputy Principal, the reference sample laboratory, the Crown contracts department, and the research laboratory. The reference sample laboratory, which is typical of most of the rooms as regards arrangements and fittings, may perhaps be described here. The tops of the working benches are of Honduras mahogany, the cupboards beneath them of oak; the bench reagent shelves of plate-glass on gun-metal brackets padded with indiarubber. Each bench is fitted with a metal filter-pump and manometer, a sink for the reception of dirty waste liquid, and a pipe leading to the reservoir under the basement, for conducting away clean used water. On one of the walls is placed a steam drying oven of copper packed in asbestos and cased in oak, and fitted with plate-glass doors. Orifices at bottom and top allow of the passage through it of a current of air which is first heated by being caused to pass through pipes surrounded by the hot packing. The waste steam escaping from the oven is led through a worm lined with pure tin contained in a drum through which cold water circulates, and the resulting distilled water for ordinary purposes collected in a

large earthenware jar fitted with gauge and overflow-pipe. The water used for this condensation and all similar purposes is returned to the clean water reservoir. Beneath the drying oven stands an oak chest which carries at its top a small copper tank, tin-plated inside, and having a tray-shaped copper top. The heated water formed by the condensation of the steam in the pipes of the drying apparatus falls into this tank, and thus yields a supply of hot distilled water, while the hollow cover serves as a bath for heating at moderate temperatures. The cupboard below is used for drying towels, dusters, &c. The floors of the evaporating cupboards and draught chambers are of slate, the top and sides being of plate-glass in oak frames; the fronts open by sliding upward, and are each connected with a counterpoise by a chain passing over a block. Each chamber is connected with the ventilating system by an air passage in the wall of the room. The evaporating apparatus consists of a copper cone sunk in the slate slab, and fitted at its upper part with a removable porcelain collar. This carries a cover of heat- and acid-resisting material perforated with holes of different sizes for the accommodation of basins and capsules. Two holes are pierced at opposite sides of the cone at slightly different heights, the higher being an inlet for steam. This, condensing, fills the cone to the level of the lower orifice, and the water thence runs to a gauge arranged so as to maintain a constant height of water in the cone. Should it for any reason be found impracticable to employ steam, the copper cone can be directly heated by a Bunsen burner fixed beneath it, any loss by evaporation being automatically replaced from the gauge, which is connected both with the water supply and waste cistern. An oak cupboard with top and front of plate-glass contains standard solutions. The burettes are fixed on a brass rack immediately above, and are connected by indiarubber tubes to the bottles containing the standardised reagents; they are filled from the stock supply by suction, the ingress of CO_2 , &c., being guarded against by bulb-tubes filled with soda-lime.

In addition to the apparatus already described, a counterpart of which is to be found in most of the rooms, the Reference Sample Laboratory is also provided with a hot cupboard with sliding plate-glass doors, and a hot trough for melting and filtering butters and fats—a class of substances largely dealt with in this room.

The Crown Contracts Department has for its work the examination of samples submitted on tender for supplies to various Crown Offices and their comparison with the actual deliveries. Besides the fittings common to all the rooms the one allotted to this work has an automatic gas-blast for long-continued ignitions, muffle gas-furnaces for similar purposes, self-regulating hot-air baths, and a sulphuretted hydrogen cupboard with a ventilating passage entering directly into the boiler flue.

The Research Laboratory is fitted similarly with the addition of a hood for combustion work.

Adjacent to the Principal's room is a Reference Library.

On the first floor are the main laboratory, the tobacco laboratory and incinerating room, and the Superintending Analyst's rooms. The former, the largest room in the building, is used for work connected with the beer and spirit duty samples. Leading out of it are a polarimeter room and a cold-storage room, the latter having walls of some 12 in. thick, containing tanks of cold brine and cased with non-conducting material. The water used in the main laboratory itself for condensing purposes is the cooled supply already mentioned: it is kept at a certain fixed temperature convenient for the work.

The fittings of the tobacco laboratory are in the main similar to those of the other rooms, with the exception that extra accommodation for microscopical work is provided. In the incinerating room there is a new device for the first ignition of the samples. This consists of a compact arrangement of small Bunsen burners, the height of the flame of each being independently regulated from

the front. The platinum vessels used are oblong in shape and are supported on a frame of cobalt iron. This material is also used for the inner shell of the muffle furnaces, which are somewhat broader and flatter than is usual. There are three ovens for moisture estimations. These are of the type in general use in the laboratory, but have in addition packed shelves through which the air current passes, entering and leaving them at opposite ends so as to ensure complete circulation in every part of the drying space. As, also, it is necessary for the temperature of these ovens to be maintained throughout the night, they are heated by steam supplied by a self-filling and self-regulating gas-boiler.

Two small staircases lead upwards from the first floor; the one to the photographic dark room, the other to the museum, to the room in which instruction in chemical matters of interest from the Revenue point of view is given to the Supervisors of Excise, and to the typists' and copyists' room. Cloak-rooms on each floor, and a goods lift fill the remaining available space.

THE DETERMINATION OF UNSAAPONIFIABLE OIL IN GREASES WITH A LIME BASE.

By HENRY BAILEY, F.C.S.

THE determination of unsaponifiable oil in greases whose base is an insoluble soap, by the usual method of complete saponification with caustic alkali and extracting the unsaponified oil from the dried soap with a suitable solvent, has, in the hands of the writer, yielded results which at best are unsatisfying, owing to the prolonged drying required to free the soap from water, and the difficulty of completely extracting the unsaponified oil from the pulpy mass.

The following scheme, in which the base is separated from the assay in an early stage of the operation, yields results of maximum accuracy in a minimum of time.

Ten grms. of the sample are weighed accurately into a beaker of about 6 ounces capacity, and boiled with water to which an excess of hydrochloric acid has been added, with constant stirring until the grease is completely decomposed and free from lumps. When decomposition is judged to be complete, the floating oils and fatty acids are separated from the acid solution by filtering through a close-pored filter-paper which has been previously moistened with water, and washed with boiling water until the washings are free from hydrochloric acid. In some cases the filtrate may have a slight iridescent appearance, but the quantity of oil sufficient to cause this is so small that it may safely be ignored. The apex of the filter-paper is now punctured, and the liquid oil washed back into the beaker by a very fine jet of boiling water. About 2 grms. of caustic potash in concentrated solution is now added, together with a little alcohol, and the solution heated with constant stirring until saponification is complete, preferably with the addition of a little more alcohol to replace the solution as it evaporates.

Evaporate most of the alcohol, add about 100 c.c. of water, and warm until clear. The solution is now transferred to a separator, any oil adhering to the beaker being washed in by means of a jet of boiling water: cool, when cold add 30 c.c. of methylated ether or petroleum spirit; shake well, and allow to stand. When the line of demarcation between the aqueous and the ethereal solutions is indistinct, the cautious addition of a few drops of alcohol will improve matters.

Draw off the soap solution, and wash the ethereal solution two or three times with water, adding the washings to the soap. Transfer the ethereal solution to a small tared wide-mouthed flask. The soap solution is replaced in the separator, and again shaken with 20 c.c. of ether

to completely free it from an oil that may have escaped extraction, and the washed ethereal solution added to the weighed flask containing the first extraction.

The ether is now evaporated, and the residue of oil dried in an air-bath at a temperature a little over 100° C. until of constant weight.

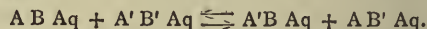
My thanks are due to Mr. J. L. Wade, of Nine Elms Lane, S.W., in whose laboratory I have experimented with this method.

THE INTERACTION OF AMMONIUM PHOSPHATE AND CORROSIVE SUBLIMATE IN THE LIGHT OF THE IONISATION THEORY OF SOLUTION.

By D. CARNEGIE and F. BURT.

LET AB, A'B', A'B, and AB' represent four soluble salts, having as their proximate components the two acid radicles A and A', and the two basic radicles B and B'.

By means of very careful measurements of physical properties, it can in general be proved that when solutions of AB and A'B' are mixed, partial mutual decomposition of the salts takes place, an equilibrium being established in which all four salts—AB, A'B', A'B, and AB'—partake.



But the cases are rare where, by purely qualitative examination involving no measurements, one can demonstrate the occurrence of chemical interaction in such homogeneous systems. As one of these rare cases may be cited the interaction of solutions of sodium chloride and copper sulphate. That solution of these salts *do* interact with the production of some copper chloride and sodium sulphate is evident from the colour change from blue to green which accompanies the addition of salt to solution of blue vitriol.

The following facts furnish a new case where interaction in homogeneous aqueous solution can be proved by purely qualitative means—a case well suited for lecture demonstration.

Neither a dilute solution of sodium phosphate (Na_2HPO_4) nor a dilute solution of ammonium oxalate will give a precipitate when added to a dilute solution of mercuric chloride; but a mixture of solutions of sodium phosphate and ammonium oxalate at once interacts with mercuric chloride, giving a dense curdy precipitate. Exactly the same precipitate is produced when ammonium phosphate solution is added to mercuric chloride. Hence it necessarily follows that solutions of sodium phosphate and ammonium oxalate partially interact with the production of sodium oxalate and ammonium phosphate.

One would off-hand expect the precipitate formed by the interaction of ammonium phosphate and mercuric chloride to be a mercuric phosphate,—to be the result of an ordinary double decomposition between the salts. But if this were the case, then sodium phosphate should act in a similar way. As we have already stated, this is not the case.

As a matter of fact, a quantitative examination showed the precipitate produced by ammonium phosphate to be infusible white precipitate, NH_2HgCl —a substance which is usually obtained as a product of the interaction of mercuric chloride and free ammonia.

Let us now glance at the whole series of these reactions in the light of the new theory of the constitution of salt solutions.

Mercuric chloride undergoes extremely little ionisation when dissolved. This follows at once from the very low electrical conductivity of aqueous solutions of the chloride. Consequently the concentration of the mercuric

ions in HgCl_2Aq is very low—so low, in fact, that the product of the concentration of mercuric ions and the concentration of $\text{C}_2\text{O}_4^{2-}$ ions from soluble oxalate solutions does not exceed the "solubility product" for mercuric oxalate. Hence mercuric oxalate cannot be obtained by interaction of dilute solutions of mercuric chloride and an oxalate.

Similar statements may be made with respect to the absence of any precipitation when solutions of mercuric chloride and any soluble phosphate other than ammonium phosphate are mixed.

When ammonium phosphate is dissolved in water, we get the ions NH_4^+ , NH_4^+ , and HPO_4^{2-} . But phosphoric acid has a very low ionisation tendency; it is, in other words, a very weak acid. Hence the HPO_4^{2-} ions combine with H^+ ions (resulting from the ionisation of water) to form undissociated phosphoric acid. More water and more ammonium phosphate undergo ionisation in the effort to re-establish the equilibrium disturbed by the removal from the sphere of action of H^+ ions and HPO_4^{2-} ions thus brought about. The end result is, that we have in a solution of ammonium phosphate a considerable concentration of free ammonium hydroxide, both in the ionised and un-ionised condition.

Similar statements might be made with regard to the solution of ammonium oxalate, with this difference, however, that oxalic acid being a much stronger—more highly ionised—acid than is phosphoric acid, the ammonium ions never reach a sufficient concentration to cause the visible formation of NH_2HgCl . Some of the NH_4^+ and Hy^+ ions do undoubtedly interact in this case, but the product of the concentration of the thus-formed NH_2Hg^+ ions and of the concentration of Cl^- ions never reaches the solubility product for NH_2HgCl .

We found that infusible white precipitate is soluble in excess of $(\text{NH}_4)_2\text{HPO}_4\text{Aq}$, though it is insoluble in excess of either NH_4OHAq or $\text{Na}_2\text{HPO}_4\text{Aq}$. In a solution of ammonium phosphate there must be more free phosphoric acid than in a solution of sodium phosphate of about equal molecular concentration. For ammonia, unlike soda, being a very weak base, the OH^- ions of the water of solution are removed from the sphere of action as undissociated NH_4OH molecules. Hence the concentration of H^+ ions must form by far the more considerable factor in the solubility product for the water which holds the ammonium phosphate in solution. But this means the existence in ammonium phosphate solutions of a comparatively large amount of phosphoric acid.

We therefore attributed the solvent action of solution of ammonium phosphate on white precipitate to the phosphoric acid which the former contains. This being so, white precipitate should dissolve readily in concentrated solutions of phosphoric acid. We found this to be the case, and the beautiful crystals which separate from the phosphoric acid solution are now under investigation.

As the concentration of aqueous salt-solutions rises, the ionisation becomes absolutely less, but the concentration of the ions becomes greater. And as chemical interaction is a function, not of the absolute amounts of the reactions but of their concentrations, we concluded that it might be possible to prepare a mercuric phosphate by precipitation from saturated solutions of mercuric chloride and ammonium phosphate. The dark red precipitate which forms when such solutions are mixed is now undergoing examination. All that can be said at present about it is, that it contains a mercuric phosphate.

Quite apart from the possible establishment of the existence of a new, and probably quite worthless, phosphate of mercury, this precipitate demands careful examination. For if, after long washing, it still be found to contain chlorine, it is clear that in this case (and perhaps in others also) another explanation of its presence lies to hand than the customary one given in such cases, viz., co-precipitation by adsorption.

Clifton College.

LABORATORY EXPERIMENTS.

By J. THOMPSON.

ONE of the simplest experiments to be performed by students of elementary chemistry is the determination of the combining proportion of magnesium and oxygen.

As the magnesium is generally burnt in a limited supply of air, the products of combustion are grey. If a fairly large quantity of this grey substance be exposed to the air for a short time, it will be found to give off the characteristic odour of ammonia.

The grey substance is a mixture of the oxide and the nitride of magnesium. Water vapour acts upon the latter, with the production of ammonia and magnesium oxide.

Platinum and tin, in the proportion of 10 of the former to 1 of the latter (by weight), form a very brittle alloy, giving off a very large quantity of heat. The combination takes place at a red heat, and the globule formed quickly becomes nearly white hot.

SEPARATIONS WITH ALKALINE ACETATES.

By HARRY BREARLEY.

(Continued from p. 167).

IV.—Chromium from Iron.

THAT in ordinary acetate precipitations chromium, if present as a chromic salt, would go down with the iron, is accepted without question. Even with minimum acetate and 10 c.c. of free acetic acid the chromium is almost completely precipitated with the iron; and yet the hydrate, as well as the acetate, of chromium is easily soluble in dilute acetic acid.

There are two phases of the acetate precipitation which, it was thought, might give at least a partial separation. These are—(a) when no free acid is present and barely enough acetate to precipitate the iron, and (b) when large volumes of acetic acid are used with minimum acetate.

The first case was tested by neutralising a mixture of ferric and chromic chlorides, diluting, and heating. At 90°C . there was no sign of turbidity; near the boiling-point the turbidity formed and deepened as the boiling was prolonged, until nearly all the iron was precipitated. Only traces of chromium were to be found in the filtrate. The mixture, as in succeeding cases, contained 1 grm. of iron and 0.1 grm. of chromium. The second case was tested by preparing solutions—both T.H. and N.H.—and precipitating in presence of 30, 50, 70, 100 c.c. acetic acid. The percentage recovery of chromium from the filtrate ranged from 3.75 to 77.5. With other degrees of dilution these results might be improved. With larger amounts of acid, if an inference from the general trend is admissible, a perfect separation might be approximated. But, however possible the separation might thus become, there are inherent difficulties which would prevent the operation ever becoming practicable.

The chief drawback, apart from the expense of the reagents, is the colour of the filtrate. This with the better separations was a deep reddish purple—so deep that in a half-litre flask it was almost opaque. It will readily be seen that on this account the precipitate can only be distinguished from the filtrate by careful examination, and so faulty filtration would often go undetected. Nor would faulty filtration be an unlikely occurrence, because the iron precipitate is so finely divided that no paper I have seen and only a carefully made asbestos filter can completely retain it. The desire to estimate chromium gravimetrically is the chief reason for seeking its separation from iron. In this case that operation would be greatly complicated by the passive nature of chromium acetates formed in

this manner (see CHEMICAL NEWS, xlviii., 114, Reinitzer; also "Bowman's Practical Chemistry," 1878 edition).

The results quoted above were obtained by oxidising with permanganate and titrating with ferrous-ammonium sulphate and bichromate.

The separation of chromium and iron, after oxidising the former to chromic acid, is attributed to Gibbs and is well known. Enquiries lead one to conclude that it is rarely used in steel works' laboratories, and it is rarely seen in text-books. The only recent instance that I know of, having made no special search, in published methods of iron and steel analysis is to be found in CHEMICAL NEWS, lxvii., 307 (Parry and Morgan). However, it still holds a place in "Select Methods," and thence one might infer that it was at least approximately accurate. I do not mind confessing that I was disappointed to find the method so unsatisfactory, until I reflected that it was no part of my endeavour to satisfy expectations. The avowed principle of the reaction is indeed a prepossessing one. The method, briefly described, is to neutralise, add acetate, oxidise with bromine or chlorine, and boil, whence the iron is precipitated, and the excess of oxidant eliminated simultaneously.

Naturally, the usual amount of acetate, hydrate, and acid were first tried. The tests throughout were made on mixtures of an acidified solution of ferric chloride and potassium chromate (1 grm. Fe, 0.1 grm. Cr). The first few separations were consistently very bad ones (percentage recovery, 26 to 27). It was thought that the presence of a strong oxidant throughout the precipitation was the condition wanting; considerable bromine was therefore added to a mixture, but the result showed no material improvement. Several repetitions of the first experiment emphasized the surprisingly low percentage recovery and drew attention to the unusually low turbidity temperature—50° to 60° C., instead of the expected 80° to 90°. Corresponding results were obtained when the chromate was replaced by bichromate, or the mixture by a sample of chromium steel.

The low turbidity temperature suggested that something in the solution was acting along with the acetate as a precipitant, and that this something could be made to act alone under suitable conditions. The mixture of iron and chromate, therefore, was neutralised, &c., as usual, except that no acetate whatever was added. The solution became turbid at 80° C., and the precipitate and filtrate had much the usual appearance. Less chromium was recovered than in any previous experiment. In a precisely similar experiment, except that the possible formation of acetates was avoided by replacing the 10 c.c. acetic acid by 10 c.c. normal hydrochloric acid, the turbidity appeared at 87° C., and the chromium in the filtrate was less than before. Attempts were made to completely precipitate the chromate by having present more ferric chloride than could be precipitated at boiling heat, and then adding dilute acetate until the precipitation was just accomplished. An impression that the chromate was much the more energetic precipitant of the two suggested this course; it was not successful.

It may be desirable to do more than merely record these observations; here and now, however, it would interfere too much with the purpose of these papers. But it is neither premature nor out of place to suggest that the deficient recovery and the allied low turbidity temperatures are due to a reaction between the chromic acid and the ferric chloride, in which an insoluble chromic compound is formed. "Watts' Dictionary" notices two ferric chromates—a basic salt, $\text{Fe}_{23}\text{CrO}_{4.2}\text{Fe}_2\text{O}_3$, formed by acting with K_2CrO_4 on iron-alum solutions; and an acid salt, $\text{Fe}_{23}\text{CrO}_4.\text{CrO}_3$, said to be formed by digesting CrO_3 Aq with $\text{Fe}_2\text{O}_3\text{H}_6$. If the said compound is either, the probability is that it is the former; anyway, it is a compound of iron with chromic acid, because if the total precipitate be dissolved in dilute hydrochloric acid, and the chromium estimated by Penny's process, as much is

found as makes, with that obtained from the filtrate, an amount exactly equal to that introduced.

Such a method as the preceding can by no stretch of favour be classed as a quantitative one. It is capable of better behaviour surely, else how came it to move amongst "select" methods. It may be noticed on re-reading the instructions that an excess of acetate is recommended. Whether this is an express statement or the customary formulæ for acetate precipitations it is difficult to say, but the word "excess" is so important that it should have been printed in italics and then qualified by "very large." I cannot resist the opportunity of repeating that in the separation of nickel and cobalt from iron this course has been adopted. The same eminent metallurgical chemist dismisses the separation of iron and chromic acid with "an excess of sodium acetate" in ordinary type.* This point strongly emphasizes the need of scrutinising methods before adopting them, and the shallowness of the economy which imagines that laboratory operators are dead losses if not producing long rows of results.

It must be admitted that with chromium steels containing only from several tenths to 2 or 3 per cent much better results are possible than I obtained with the previously-mentioned proportions, else such a deficiency could not have gone undetected in the most ill-conditioned laboratory. Where the method has been tested on armour plate borings, with strict adherence to instructions, I am informed that some 50 to 70 per cent of the contained chromium has been estimated. This may readily be explained. The less the percentage of chromium, the greater the proportion of acetate to chromate in the oxidised solution, and the greater the proportion of acetate to chromate (?) precipitated. If this explanation be true, it should happen that all else being equal, the greater volume of acetate should accompany the greater percentage recovery. In Table XIV. this is shown to be so.

TABLE XIV.

Volume of strong acetate. C.c.	Per cent recovery with—	
	Ammonia acetate.	Soda acetate.
10	43.6	50.5
50	47.2	70.0
100	72.0	—
200	81.5	—

The amounts of iron and chromium are as before. The solutions contained total hydrate and 10 c.c. acetic acid. The acetates previously used are too dilute to use in this case, within the limit of our standard volume (1000 c.c.). They were therefore made about 13 times as strong as usual, e.g., the ammonium acetate is approximately that made by neutralising 33 per cent acetic acid with 880 ammonia.

The effect of increasing volumes of acetic acid is to give higher results, but only so in a slight degree.

The foregoing may help to explain why the acetate separation of iron and chromium has fallen into disuse. With such modification as we are now able to make, it is scarcely worth while to formulate the method anew. If there are ever circumstances which make the method a desirable one, well. For determining chromium in iron and steel such methods as Galbraith's, or Stead's modification of it, are all that can be desired, and if a gravimetric method were needed, the separation in question is almost as troublesome and less accurate than fusion with alkalis.

After oxidising the chromium, the acetate is frequently replaced by a pure or carbonated alkali. A few tests rapidly made tend to show that in such cases a similar error—though smaller—is introduced, and may, similarly, be more and more nearly eliminated by adding increasing

* It is only fair to say that the author of these instructions says at the same time "a satisfactory method for the determination of chromium has yet to be devised."

quantities of alkalis. It may be noteworthy, too, that aluminium and chromium are separated under precisely the same conditions as iron and chromium (see "Fresenius," 7th edition, page 428), and a corresponding basic salt, $\text{Al}_2\text{CrO}_4 \cdot 2\text{Al}_2\text{O}_3 \cdot 21\text{H}_2\text{O}$, is said to be formed in a like manner. This makes it desirable to examine the separation of these two elements. Quite casually, too, I learn from an abstract in the *Journ. Chem. Soc.* that Marchal and Wiernik (*Zeit. Angew. Chem.*, 1891, 511—513) claim to have obtained perfect separations of iron and chromium by oxidising the chromic salt with freshly-precipitated manganese dioxide. Thus it appears that there are a number of methods over which suspicion is cast. If I may reserve this study to myself, it shall not be neglected when "Separations with Alkaline Acetates" have been completed. There is evidence that the use of an alkaline chromate will effect separations which with alkaline acetates are impossible.

(To be continued).

SOME NOTES ON CONCENTRATED SOLUTIONS OF LITHIUM AND OTHER SALTS.*

By JOHN WADDELL, B.A., D.Sc., Ph.D.

IN an article published nearly two years ago in the *CHEMICAL NEWS* (lxxii., p. 201), I gave a record of experiments on the relative amounts of water absorbed by the molecular weights of lithium nitrate and calcium nitrate when they were enclosed in the same bottle along with a limited quantity of water. Each of the salts and the water were in tubes like small test-tubes, the quantity of water being small, so that the solutions of the lithium and calcium salts were concentrated. It was found that the lithium nitrate absorbed more water-vapour than the amount calculated on the assumption that lithium nitrate and calcium nitrate are dissociated equally; the indication being, that while neither of the salts is completely dissociated in concentrated solutions, lithium nitrate is more largely dissociated than calcium nitrate. Lithium nitrate was also found to be dissociated to a greater extent than potassium, strontium, or barium, nitrate. It was mainly with a view to seeing whether lithium chloride and sulphate would show similarly greater dissociation than other chlorides and sulphates that the few experiments described in these notes were undertaken.

The experiments are not all complete, the weight of the tubes not being in every case constant; but they have been carried on long enough to make the general conclusions pretty certain, and the results are therefore given without longer delay.

Lithium, strontium, barium, and potassium chlorides were investigated with the results given below:—

Table of Quantities of Water in Grms. taken up by the Molecular Weight (in M.grms.) of the Chlorides.

1 LiCl ..	0.709	0.580	0.597	0.401
1 BaCl ₂ ..	0.966	0.807	—	—
1 SrCl ₂ ..	1.021	—	—	0.604
1 KCl ..	—	—	0.513	0.323

The figures in the last column show that while potassium chloride is not so largely dissociated as lithium chloride, strontium chloride is dissociated to a greater extent. The only alternative to this last conclusion is that both the lithium chloride and the strontium chloride are completely dissociated—a conclusion which is not only *a priori* unlikely, but is negated by the results given in the first column, because there it is seen that the water

absorbed by the lithium chloride is more than two-thirds as much as that absorbed by the strontium chloride, which would seem to prove that the rate of dissociation of the former salt is increasing as compared with the latter. If two-thirds of the lithium chloride and three-fourths of the strontium chloride were dissociated, the number of ions in the one case would be just two-thirds of that in the other; or the same result would follow if one-third of the lithium chloride and one-half of the strontium chloride were dissociated. These are two out of an indefinite number of solutions of the problem, so that it is evident that the experiment does not give sufficient data for determining to what extent the various salts are dissociated.

The amount of water absorbed by the barium chloride is not far short of that absorbed by the strontium chloride, so that it too is probably more dissociated than the lithium chloride.

In the case of the sulphates, lithium was compared with magnesium and zinc. The last two were weighed out in the form of $\text{MgSO}_4 \cdot \text{H}_2\text{O}$ and $\text{ZnSO}_4 \cdot \text{H}_2\text{O}$, that this was their composition being proved by precipitating a portion of the salts with barium chloride.

As the tubes have not attained constant weight, the last two weighings are given, so that it may be seen which tubes were gaining weight and which losing, and at what relative rate.

Table of Quantities of Water in Grms. taken up by the Molecular Weight (in M.grms. of the Sulphates). Those marked + were increasing, those marked — were decreasing.

1 Li ₂ SO ₄ ..	1.108	1.114+	1.790	1.797+
1 MgSO ₄ ..	0.693	0.687—	1.078	0.067—
1 ZnSO ₄ ..	0.620	0.616—	1.070	1.053—
1 Li ₂ SO ₄ ..	—	—	1.924	1.925

The magnesium sulphate is slightly, but only slightly, more absorbent than the zinc sulphate. That the difference of absorption is not large may be seen by noticing that where the magnesium and zinc salts had nearly equal quantities of water, the zinc sulphate was losing more rapidly than the magnesium sulphate; but when the zinc sulphate had only six-sevenths as much water as the magnesium sulphate, the zinc sulphate lost more slowly. The lithium sulphate was far more absorbent than the other two, and plainly—if dissociation into ions is indicated by absorption—the lithium salt is considerably more dissociated than the others.

There were also some further experiments with nitrates, and, in order to show in what direction the action was going in the cases in which the condition was not constant, or so nearly so that the difference in six or seven days was not noticeable, a plus or minus sign is given, with the increase or decrease in the time mentioned.

Table of Quantities of Water in Grms. taken up by the Molecular Weight (in M.grms.) of the Nitrates.

1 KNO ₃ ..	0.402	0.683	1.233+3	1.635—4	3.275
1 LiNO ₃ ..	0.647	0.969	1.611+4	1.968	—
1 NaNO ₃ ..	—	—	1.649	2.218—1	—
1 Ca(NO ₃) ₂ ..	—	—	1.933+2	2.081	—
1 Sr(NO ₃) ₂ ..	—	—	—	2.045	—
1 Ba(NO ₃) ₂ ..	—	—	—	—	—
1 AgNO ₃ ..	—	—	—	—	3.300

The most noticeable feature in this table is that, though potassium nitrate is not so absorbent, and therefore presumably not so much dissociated as lithium nitrate, sodium nitrate, in both cases, observed, has taken up more water. Strontium and calcium are not far apart when the dilution is that given above; the difference in more concentrated solutions, as I showed in my former paper, is greater. The barium nitrate weighings are not given, because in the first case water had been added in

* Read before the British Association (Section B), Toronto Meeting, 1897.

large quantity, and all of the gain in the other tubes came from it; in the second case the tube contained crystals.

In the comparatively dilute solutions of potassium and silver nitrate, the absorption was practically equal. Along with these solutions two tubes containing mercurous nitrate were introduced, in order if possible to see whether the formula $\text{Hg}_2(\text{NO}_3)_2$ or HgNO_3 would correspond best with the result obtained, but the absorption of vapour by the salts was so slow that they were not brought into solution, and so nothing could be determined regarding the formula.

I described, in the former paper, some experiments with potassium chloride, bromide, and iodide, which proved that the invaporation of these salts, provided there is enough water present to bring them all into solution, is approximately equal. I have since compared potassium chloride and potassium nitrate, and, as the table shows, the chloride is very considerably more absorbent.

Table of Quantity of Water in Grms. taken up by the Molecular Weight (in M.grms.).

1 KCl	0.641	0.902
1 KNO ₃	0.482	0.780

The dissimilarity between the chloride and nitrate is all the more remarkable because they agree much more nearly as to solubility than the chloride does with the bromide and iodide. If the first table containing the chlorides, and the table containing the nitrates, be referred to, it will be seen that lithium and potassium chloride differ less in the amount of water absorbed (or, in other words, have more nearly the same vapour pressure for the same strength of solution) than lithium and potassium nitrate. Indeed, though with the concentrated solution that I had, the lithium chloride is more dissociated than the potassium chloride; the table of electrical conductivities given by Kohlrausch indicates that if the dilution were twice as great the potassium chloride would be the more dissociated.

The conclusion to be arrived at from my experiments is, that there is no special peculiarity belonging to lithium salts as regards invaporating power, there being some chlorides and some nitrates that absorb more water per molecule.

The behaviour of salts in the concentrated solutions is so haphazard that there is little more to be done with them, and I should not consider the investigation worth pursuing further in this direction. The method is, however, adapted for comparing the vapour pressure of different solutions. For example, if it is desired to show the student that molecular weights of such salts as potassium chloride and potassium iodide, having equal quantities of water, have the same vapour pressure, it would merely be necessary for him to make two such solutions, and arrange three experiments in one of which the solutions are used as prepared above, a second in which the potassium *chloride* is made slightly more dilute, and a third in which the potassium *iodide* is made slightly more dilute. These three bottles with the enclosed tubes may be set aside for a few days, and then on weighing there will be found to be no change in the tubes in the first bottle; in the second, the tube containing potassium chloride would be found to be losing weight and the iodide gaining; while in the third, the reverse action would be seen to be going on.

In this way the well-known law of vapour pressures could be illustrated without any elaborate apparatus. Moreover, if a number of experiments are to be performed, it is easier to set them up and allow them to stand for a few days than to go on with a series of determinations of vapour pressure in an apparatus which must be carefully cleaned and dried each time.

Why my experiments took so long was because I was working at the invaporation from the beginning, and did not make use of solutions made up to a suitable strength, my purpose being different from the one of which I am

now speaking. As I have stated, there is not much regularity in the concentrated solutions, no law having been found by which it would be possible to predict which of two concentrated solutions would have the greater vapour pressure; but this method of comparing vapour pressures may sometimes be found valuable.

Kingston, Canada.

THE TITRATION OF SODIUM THIOSULPHATE WITH IODIC ACID.*

By CLAUDE F. WALKER.

THIS investigation was undertaken to determine the nature and limitations of the reaction between iodic acid and thiosulphuric acid, and to show the expediency of employing iodic acid in standard solution for the direct titration of sodium thiosulphate. Riegler states that iodic acid is readily obtained pure, and that a standard solution can be kept a long time unaltered. He further states that when a solution of sodium thiosulphate is titrated with iodic acid the reaction takes place according to the equation—



Under which circumstances no free iodine will be evolved until all the sodium thiosulphate has been oxidised to tetrathionate. The first drop of iodic acid in excess, however, will react with the sodium iodide that has been formed and liberate iodine, as shown by the equation $5\text{NaI} + 6\text{HIO}_3 = 5\text{NaIO}_3 + 3\text{H}_2\text{O} + 3\text{I}_2$, thus furnishing an accurate means for determining the end of the reaction.

A careful repetition of Riegler's work shows that his conclusions are to a great extent erroneous. Thus, "chemically pure" iodic acid is very likely to contain too much iodine, due probably to the presence of the anhydride. Riegler's proposed method of titration depends on two different reactions, and these must be definite, complete, and non-reversible under the conditions of the analysis. Thus one molecule out of every six of iodic acid should be reduced by six molecules of thiosulphate, forming a neutral mixture of iodide and iodate, when it might be expected that iodine would be liberated by the first trace of iodic acid in excess. There was striking evidence, however, of some obscure reaction of the thiosulphate, which influences the reduction of the iodic acid, so as to make it impossible to calculate analyses according to Riegler's reaction. It is not impossible that some third unstable compound of iodine may be formed as an intermediate product and delay the titration of iodine.

It appears, however, that Riegler's proposed process for standardising sodium thiosulphate, is impracticable unless it can be so modified as to do away with a number of sources of error.

The analyses of solutions of iodic acid in this work was invariably performed by adding to the portion of the solution to be analysed an excess of potassium iodide, acidifying with 5 c.c. of dilute (1:3) sulphuric acid, and recovering the liberated iodine by titrating the acid solution with sodium thiosulphate, or by neutralising with an excess of potassium bicarbonate, and titrating the alkaline solution with arsenious acid. In either case one-sixth of the iodine recovered was calculated to iodic acid, according to the equation $5\text{HI} + \text{HIO}_3 = 3\text{I}_2 + 3\text{H}_2\text{O}$.

In the present work it was found convenient to analyse the iodic acid in quantities of about one-tenth of a grm., when the variation in results in the same series is inappreciable.

By blank experiments it was found that one drop of iodine was necessary to bring out the starch blue, so this was uniformly applied in the analytical work. Two dif-

* Abridged from the *American Journal of Science*, vol. iv., Sept., 1897.

ferent samples of "chemically pure" iodic acid were used to determine whether its purity is sufficient to admit of its direct application in standard solutions. Quantities of both these were dried over sulphuric acid to constant weight. A third sample was prepared by dissolving the purest obtainable iodic anhydride in water and evaporating at ordinary temperature.

The resulting crystalline mass was dried over sulphuric acid for a week, until ceasing to lose weight; it was presumed to consist of the pure normal acid. Two presumably decinormal solutions of each of the first two samples, and one such solution of the third sample of iodic acid, were made by dissolving 17.585 grms. in 1 litre of water at 15° C. Analyses of these solutions gave the following results:—

Solution.	Sample used.	HIO ₃ taken. Grm.	HIO ₃ found. Grm.	Error. Grm.
I.	A	0.1055	0.1066	0.0011+
II.	A	0.1055	0.1062	0.0007+
III.	B	0.1055	0.1065	0.0010+
IV.	B	0.1055	0.1073	0.0018+
V.	C	0.1055	0.1053	0.0002—

These results, which are averaged from a large number of determinations, show that while the deviation from the theoretical strength of the solution in the case of the acid prepared from anhydride is hardly appreciable, the solutions made from the purchased product contain a very appreciable amount of iodine in excess of the theoretical quantity.

To determine whether a solution of iodic acid, once prepared and standardised, will retain its strength for a long time, two such solutions were kept for four months (in the dark) and then again analysed. The results (averages of several determinations) given below substantiate Riegler's observation that a solution of iodic acid will remain of constant strength.

Constancy of Strength of Iodic Acid Solutions.

Iodic acid solution.	First analysis. HIO ₃ found. Grm.	Second analysis (after four months). HIO ₃ found. Grm.	Variation. Grm.
I.	0.1073	0.1072	0.0001—
II.	0.1049	0.1046	0.0003—

A series of analyses made by oxidising the sodium thiosulphate to sulphate and precipitating and weighing as barium sulphate, gave results identical with those obtained with iodine; proving that all the sulphur present was in the form of thiosulphate.

According to Riegler's equation, sodium thiosulphate and iodic acid react molecule for molecule, and solutions of them should therefore require for their mutual saturation volumes inversely proportional to their concentration. It was found, however, that when a one-twentieth normal solution of sodium thiosulphate was titrated in the presence of starch with an approximately decinormal solution of iodic acid, a distinctly blue colour was produced long before the theoretical amount of iodine had been added. It was further noticed that the end-point of the reaction was far from distinct; a faint tint of blue at first being visible, then suddenly becoming deeper, and immediately reappearing when bleached with sodium thiosulphate. It was found, however, that the addition of a considerable quantity of potassium iodide to the solution, either before or during the titration, had the marked effect of making the reaction sharp and distinct, entirely preventing the "after separation" of iodine, and at the same time postponing the appearance of the starch-blue until a quantity of iodic acid had been added considerably in excess of the theoretical. The experiments were executed with entirely different reagents, and under varied conditions of concentration, the results in every case confirming those already observed.

For the purpose of more particularly investigating this subject, there were prepared and standardised an approximately decinormal solution of sodium thiosulphate and an approximately one-fiftieth normal solution of iodic acid. Measured portions of the sodium thiosulphate solution were titrated with the iodic acid in presence of starch under varying conditions of mass, time, and dilution.

To determine the variability of the end-point of the reaction, a series of experiments was made. Measured amounts of the thiosulphate solution were drawn into an Erlenmeyer beaker, 5 c.c. of starch were added, and the iodic acid slowly dropped in, with constant agitation, until the first blue tint appeared. The results obtained were as follows:—

Variation of the End-reaction between N/10 Sodium Thiosulphate and N/50 Iodic Acid in the Absence of Potassium Iodide.

	Na ₂ S ₂ O ₃ taken. C.c.	HIO ₃ added. C.c.	Mean value. C.c.	Variation. C.c.
1.	6	28.13	28.32	0.19—
2.	6	27.79		0.53—
3.	6	28.03		0.29—
4.	6	28.32		0.00
5.	6	28.32		0.00
6.	6	28.71		0.39+
7.	6	28.83	18.68	0.51+
8.	6	28.43		0.11+
9.	4	18.94		0.26+
10.	4	18.67		0.01—
11.	4	18.50		0.18—
12.	4	18.60		0.08—

These experiments indicate that the constancy of the end of the reaction in different titrations of equal volumes of the same solution depends to a certain degree on the volume of sodium thiosulphate taken. The probable error which these irregularities would introduce in any series of practical analyses by this method is obviously greater than can ordinarily be permitted in iodometric work.

The experiments in the following table were performed in exactly the same manner as those in the last series, except that 2 grms. of potassium iodide were added to the sodium thiosulphate before titrating.

Variation of the End Reaction between N/10 Sodium Thiosulphate and N/50 Iodic Acid in the Presence of Potassium Iodide.

	Na ₂ S ₂ O ₃ taken. C.c.	HIO ₃ added. C.c.	Mean value. C.c.	Variation. C.c.
1.	6	32.53	32.48	0.05+
2.	6	32.45		0.03—
3.	6	32.67		0.19+
4.	6	32.37		0.11—
5.	6	32.36		0.12—
6.	6	32.50		0.02+
7.	4	22.30	22.19	0.11+
8.	4	21.98		0.21—
9.	4	22.17		0.02—
10.	4	22.30		0.11+

These experiments show that in the presence of iodide of potassium the end reaction is practically independent of the amount taken for analysis. It is therefore evident, from studying the above figures, that the presence of potassium iodide in the sodium thiosulphate to be titrated will bring the variation of the formation of the reacting tint within permissible limits.

Another series of experiments was made to determine the nature and effect of the "after colouration" observed to take place when a solution of sodium thiosulphate, free from potassium iodide, was titrated with iodic acid to blue colouration and then bleached with sodium thiosulphate. The results are given below:—

Effect of Dilution and Lapse of Time on the
"After Colouration."

	Na ₂ S ₂ O ₃ taken. C.c.	HIO ₃ added. C.c.	Na ₂ S ₂ O ₃ added. C.c.					Vol. C.c.
			15 mins.	45 mins.	1 hr. 45 mins.	2 hrs. 45 mins.	20 hrs.	
1.	6	27'68	0'25	0'13	0'08	0'00	0'03	0'49
2.	6	27'70	0'20	0'10	0'03	0'03	0'03	0'39
3.	6	28'17	0'16	0'10	0'03	0'01	0'00	0'30
4.	6	27'03	0'60	0'26	0'09	0'03	0'00	0'98
5.	6	27'60	0'93	0'28	0'06	0'04	0'04	1'35
6.	6	28'60	1'34	0'46	0'17	0'03	0'14	2'14
7.	6	28'85	1'20	0'50	0'28	0'06	0'27	2'31
8.	6	31'63	1'46	0'74	0'10	0'21	0'23	2'74
9.	6	29'90	1'04	0'60	0'23	0'15	0'46	2'48
10.	6	36'09	1'60	1'23	0'63	0'34	0'18	3'98
11.	6	37'59	1'65	1'33	0'72	0'27	0'10	4'07
12.	6	37'23	1'92	1'05	0'64	0'33	*	300

* No observation.

In the experiments with small volumes, the evolution of iodine in any quantity ceased after two or three hours, though the solution would become coloured as often as it was bleached for a number of days. The traces of iodine thus set free were, however, only equivalent to one or two drops of sodium thiosulphate, but the larger volumes continued to liberate iodine in abundance for a very long time. The amount of iodine thus liberated after the first colouration evidently varies with the amount of iodic acid required for the titration; both of these quantities increase at a regular rate with the volume of the solution.

To try with what accuracy the reaction between sodium thiosulphate and iodic acid may be applied to the direct estimation of one of these substances by the other, the averaged results of a large number of titrations are here given. The operations were conducted as directed by Riegler, equal volumes of standard thiosulphate being titrated with iodic acid of known strength in the presence of starch, and under different conditions of time, dilution, and mass, the volume of iodic acid required being in each case compared with the volume theoretically required by Riegler's equation.

Titration of N/10 Sodium Thiosulphate with N/50
Iodic Acid.

	Na ₂ S ₂ O ₃ taken. C.c.	HIO ₃ added. C.c.	HIO ₃ required by theory. C.c.	Error.		Present. Grm.	Vol. C.c.
				C.c.	Per cent.		
1.	4	18'68	20'32	1'64—	8'0—	—	50
2.	6	28'32	30'48	2'16—	7'0—	—	50
3.	6	27'32	30'48	3'16—	7'0—	—	150
4.	6	28'73	30'48	1'75—	6'0—	—	200
5.	6	30'77	30'48	0'29+	0'01+	—	250
6.	6	36'97	30'48	6'49+	21'0+	—	300
7.	6	27'46	30'48	3'02—	10'0—	—	50
8.	6	26'15	30'48	4'33—	14'0—	—	150
9.	6	26'50	30'48	3'98—	13'0—	—	200
10.	6	27'16	30'48	3'32—	10'0—	—	250
11.	6	32'93	30'48	2'45+	8'0+	—	300
12.	4	22'19	20'32	1'87+	9'0+	0'2	50
13.	6	32'48	30'48	2'00+	7'0+	0'2	50

* HIO₃ added until first blue colour.

† Calculated by subtracting from the amount of iodic acid originally titrated the volume of thiosulphate required to bleach the solution after standing twenty hours.

These results show plainly that the amount of iodic acid required to decompose a given amount of sodium thiosulphate may be considerably above or below that required by Riegler's equation. Thus, with small volumes, and in the absence of potassium iodide, the thiosulphate is destroyed, and the separation of iodine commences when only 93 per cent of the theoretical amount of acid has been titrated. At higher dilutions the action is retarded, so that at 250 c.c. very nearly the theoretical amount of acid is required to produce the first blue colour, and at 300 c.c. an excess of 21 per cent over the theoretic-

cal amount must be added, and it appears that for all volumes below 300 c.c. the original thiosulphate is destroyed when about 90 per cent of the theoretical amount of iodic acid has been added. Potassium iodide retards the action, so that at small volumes an excess of about 8 per cent of iodic acid must be added to completely destroy the thiosulphate and commence the separation of iodine. It is obvious from the preceding experiments that the reaction between iodic acid and sodium thiosulphate is so indefinite in its nature, and so dependent for its completeness on conditions of time, dilution, and mass, that its direct application as a means of standardising solutions must remain impracticable.

ACETYLENE GAS.

THE Explosives Department of the Home Office has recently had under consideration the question of the restrictions to be applied to the manufacture and keeping of acetylene gas, and has conducted various experiments with the object of gaining information on this matter. The results show conclusively that acetylene gas *per se*, when under a pressure of something less than two atmospheres, is violently explosive; whereas at a pressure of less than one and a half atmospheres it appears to be reasonably free from liability to explosion, provided it is not admixed with oxygen or atmospheric air.

For commercial and practical purposes it is considered sufficient to allow a pressure of 20 inches of water above that of the atmosphere (*i.e.*, roughly, about one and one-twentieth atmospheres), and it is accordingly proposed to draw the safety line at this point, and to declare acetylene when subject to a higher pressure to be an "Explosive" within the meaning of the Explosives Act, 1875.

In France and Germany the authorities have fixed the limit of danger at one and a half and one and one-tenth atmospheres respectively, and have imposed prohibitions or restrictions on the keeping or manufacture of the gas when it is at a higher pressure.

Whitehall, October 5, 1897.

NOTICES OF BOOKS.

An Electrical Method of Determining the Moisture Content of Arable Soils. By M. WHITNEY, D. GARDNER, and L. J. BRIGGS. Washington: Government Printing Office. 1897.

THE only method in general use for determining the moisture in soils is the very simple one of taking a sample of the soil from the field, at any desired depth, and drying it at 100° C.; but from a large number of observations it is found that this method is not accurate to within 2 per cent, plus or minus.

Another method which has been tried is that of burying bricks about 8 inches below the surface, and taking them up and weighing them day by day; this method is obviously of no use whatever. The possibility of using the electrical resistance of soils for the determination of moisture was suggested by the necessity of getting a good "earth" for lightning conductors, &c.

Soils are composed of fragments of various minerals and salts, all more or less soluble in water; even quartz is slightly soluble in water containing carbonic acid, as soil waters usually do, and this dilute solution conducts the electric current. The specific resistance of a solution is the resistance of 1 c.c. of liquid between two parallel electrodes 1 c.m. square and 1 c.m. apart, and the specific conductivity as used in this Bulletin is the reciprocal of the specific resistance.

The Wheatstone Bridge method was used with an alternating current and a telephone in place of a galvanometer. The electrodes finally adopted for field-work consist of carbon plates, each 3 inches long, $\frac{3}{8}$ inch wide, and $\frac{1}{8}$ inch thick, copper-plated at one end, to which a copper wire was soldered; the plates can be placed at any desired depth, but 1 to 2 feet has been found the most convenient; but we are unable to find any definite statement as to how far apart they are placed. The whole apparatus is standardised by comparing the readings with a number of observations taken by the old method, and a table of values is constructed.

An Electrical Method for Determining the Temperature of Soils. By M. WHITNEY and L. J. BRIGGS. Washington: Government Printing Office. 1897.

In perfecting the electrical method of determining the moisture in soils, a compensation cell, having the same electrical temperature coefficient as the soil, is used as one arm of the Wheatstone Bridge, in order to eliminate temperature effects in determining the electrical resistance of soils, and this cell can be very easily used for taking the temperature of the soil.

These cells are constructed of small strips of glass, cemented together with marine glue; their dimensions are 3 inches $\times$ 2 inches $\times$ $\frac{1}{4}$ inch; the electrodes consist of strips of carbon, to which wires are soldered, cemented to the glass. They are then standardised, and the resistance reduced to a common standard, viz., 1000 ohms at 60° F. on being buried, and readings taken with the Wheatstone Bridge; the temperature is arrived at by calculation.

An Electrical Method of Determining the Soluble Salt Contents of Soils. By M. WHITNEY and T. H. MEANS. Washington: Government Printing Office. 1897.

A VERY simple and delicate method has been devised for determining the soluble salt contents of soils, in samples taken from the field. The method consists essentially of mixing a known quantity of a soil with a known proportion of pure water, and determining the specific resistance; then an equal weight of the same soil is mixed with a weak salt solution equal in volume to the pure water added, and the specific resistance again taken; the amount of salt added being accurately known, and also the effect it had on lowering the resistance, it is easy to calculate the quantity of salts originally present in terms of the salt solution used.

A Treatise on Medical and Scientific Radiography. ("Traité de Radiographie Médicale et Scientifique.") By Dr. FOVEAU DE COURMELLES. Pp. 470, with 176 Illustrations. Paris: Océane Dooin. 1897.

PROFESSOR RÖNTGEN'S discovery, that the X rays emitted by a Crookes tube affected a sensitised photographic plate, is only of recent date, being first made known at the latter end of 1895; but already, in this short space of time, enormous developments have been made in the subject, more especially in connection with surgery, and the subsequent discovery of the phosphorescent screen enables us not only to photograph the internal organs, but practically to see them. A great deal has already been written on this subject, but mostly in the form of papers and articles. The large volume now before us treats of the whole subject from its beginning up to as far as it is at present understood, and the author states that if his book rapidly becomes old-fashioned he will be the more happy, as it will show that we are making further advances in our knowledge of the subject.

In Chapter I. the early experiment of the Abbé Nollet, in 1753, is first mentioned, but we rapidly come to more modern times, when the so-called "black light" of

Moser, and Breguet, the stratified discharge of Crookes, the experiments of Hertz and Lenard, and finally Röntgen's discovery, are described and discussed.

Chapters II. and III. are devoted to the enumeration of the different sources of electrical energy, electrolysis, accumulators, &c.; while in Chapters IV. and V. dynamo-electric and electrostatic machines are dealt with. We next come to currents of high frequency, radiations both invisible and of intense therapeutic action, no longer in a vacuum, but permeating the atmosphere with the greatest ease. A lot of apparatus of special forms, adapted to medical wants, is described in the succeeding chapters, together with the methods of manufacture, pumps, coils, &c.

The form of the bulbs has been varied considerably by different workers, but they may be divided into three groups,—those in which the cathodic rays act directly on the sides of the glass tube itself, those where the cathodic rays are concentrated on some sort of a mirror which reflects them, and those in which the two preceding ones are combined. The first is the most simple, but the second form is more practicable and is the one mostly used, the indirect action being much more intense, and requiring a shorter time to obtain good results.

The radioscope, which we find in Chapter XIV., enables us to see at once certain parts of the interior of human bodies, as well as through other substances, and a very good illustration shows the manner of its application. It is now being extensively used in the French hospitals, not only for examining broken bones or searching for bullets, but also for diagnosing diseases, such as pleurisy, tuberculosis, gout, rheumatism, &c., while the internal deformities in women caused by tight lacing are very easily detected. In fact, it is medical and surgical science that has benefited principally by the discovery and application of the X rays.

The volume is one of great interest and fascination; it is well printed, on good paper; it has but one fault, one very common to French books—there is no Index.

The Manufacture of Artificial Mineral Waters and other Effervescent Beverages. ("Die Fabrikation der Künstlichen Mineral Wässer und anderer Mousserende Getränke.") By Dr. B. HIRSCH and Dr. P. SIEDLER. Third Edition, with 103 figures. Brunswick: Vieweg and Sons. 1897. 8vo., pp. 386.

THE work before us is written not merely in the German language, but most decidedly from a German point of view.

The authors treat, in the first place, of mineral waters in general, of the origin and ingredients of the springs, of the changes to which they are liable, their synthesis and classification. Their classification is complicated, as it is founded both on chemical and medicinal considerations, or on a combination of these two points of view.

Thus we have here alkaline waters, sulphate of soda waters, chalybeate brines, bitter waters, sulphated waters, earthy or calcareous waters, neutral thermæ, and poor in mineral matters.

As a distinct class are mentioned semi-natural mineral springs. Here comes the admission that relatively few springs possess well-marked medicinal properties, but serve merely as a base for the addition of drugs.

There exist also "semi-mineral waters," to which extraneous matter has been added to improve the taste; the principal of these additions is carbonic acid, which is employed both as a gas and as a liquid.

The admittedly artificial mineral waters were mentioned by Pliny, and in modern times were produced by Priestley and Lavoisier, and in 1787 artificial Selzer water was produced on the large scale by Meyer of Stettin, and the manufacture is still flourishing. Some of the artificial waters consist merely of water saturated with carbonic acid, but in other cases soluble salts are

added. The greatest care is—or ought to be—used in the selection of the water. It should be colourless and inodorous, even when warmed and exposing an extensive surface. Nor should it have a sweetish, bitter, chalybeate, inky, saline savour.

It is to be noted that the non-European mineral springs have been overlooked. This is to be regretted. South Africa is exceptionally rich in mineral waters, as was shown in the Indian and Colonial Exhibition. The question arises whether the importation and sale of South African waters should not be promoted in this country, so as to take the place of those from the European Continent, which by vigorous advertising have almost secured a monopoly of the home market.

The Organised Science Series. First Stage: Sound, Light, and Heat. By JOHN DON, M.A., B.Sc. London: W. B. Clive, University Correspondence College Press. Pp. 307.

THIS book is ably and clearly written, and no one need search for errors in its teachings unless anxious to lose his labour. At the same time it is most peculiar, and we may venture to say unhappy. It is examinational to the core, and boasts of the number of Correspondence College students who have "passed" during the last five years. How many of them have made any discovery—in other words, have contributed—to the sum total of human knowledge we are not informed. We may perhaps be pardoned for doubting whether the author considers this the grand object to be aimed at by a University, or is not perhaps fully content with securing for his students exhibitions, scholarships, and "places in honours."

CORRESPONDENCE.

SUPPOSED NEW ELEMENT WITH IRON.

To the Editor of the Chemical News.

SIR,—In last week's number of the *CHEMICAL NEWS* (vol. lxxvi., p. 171) I noticed a letter from Mr. Jones, in which he suggests that the metal I separated from iron might prove to be molybdenum. The properties of the metal do certainly resemble those of molybdenum very closely—a fact of which I have been fully aware. In my first notes on the reactions of the metal I find that "a solution of the metal heated with Na_2HPO_4 and a few drops of HNO_3 produce no precipitate." How I managed to leave out this reaction in my article in the *CHEMICAL NEWS* I really do not know. The metal has been repeatedly tested since then for molybdenum, but without success.

I have during the last week gone over the reactions of the metal, the metal used being specially prepared for the purpose. I could find no traces of molybdenum, nor was I able to find any fault with the reactions of the metal as described in my article in the *CHEMICAL NEWS*.

I have also specially prepared some metal from boiler-dust; it was most carefully tested for molybdenum, but without success.

A small quantity of metal obtained from boiler-dust a considerable time ago yielded a very small quantity of molybdenum, but not sufficient to interfere with the reactions of the metal from which it was separated.

The fact that the metal is precipitated by zinc (the characteristic blue colour produced by the action of zinc on molybdenum being conspicuous by its absence), that it combines with hydrogen (producing a gas from which the metal can easily be deposited), and that it is not precipitated by Na_2HPO_4 and HNO_3 when warmed, led me to believe that the metal was not molybdenum, as these are

certainly not properties of the latter metal as far as I know.—I am, &c.,

GETHEN BOUCHER.

The Laboratory,
North Lonsdale Iron and Steel Company, Lim.,
Ulverston, Lancashire, October 6, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Moniteur Scientifique,
Series 4, Vol. xi., Part 2.

Review of Photography.—A. Granger.—According to recent information the Americans have been for some time using chromo-photography for printing advertisement posters. This has been done by means of three monochromes, and the following solutions have been recommended. For violet—7 c.c. of concentrated solution of cupric chloride, 17 c.c. of water, and 3 c.c. of ammonia; after filtration 3 c.c. of concentrated methyl violet B and 5 c.c. of fuchsine is added. For orange—15 c.c. of concentrated solution of chloride of cobalt, 35 c.c. of water, 25 c.c. of bichromate of ammonium, and 2 c.c. of ammonia. For green—a solution of sulphate of nickel. These solutions are placed in reservoirs of plate glass $\frac{1}{4}$ th of an inch thick.

New Methods of Converting Paranitrodiaminotriphenylmethanes into Fuchsines, or into Bases of the corresponding Fuchsines.—Maurice Prud'homme.—Already noticed.

Contribution to the Study of the Formation of Amylic Alcohol in Commercial Fermentations.—Lucien Gentil.—The presence of amylic alcohol in industrial fermentations has always been observed in more or less quantity, and the question of whether this alcohol was a constant product of alcoholic fermentation, as are succinic acid and glycerin, has been the subject of a good deal of discussion, and the author is led to conclude that it is not formed in a perfectly sterile medium, which is then infected with a pure cultivation of yeast.

Some Reactions of Phospham.—R. Vidal.—When phospham, PN_2H , is heated to a bright or strong red heat in the presence of alkaline carbonates the following reaction takes place: $\text{—PN}_2\text{H} + 2\text{CO}_3\text{R}_2 = \text{PO}_4\text{R}_2\text{H} + 2\text{CNOR}$. That is to say, an alkaline cyanate is formed; the introduction of a reducing agent, such as carbon, into the mixture gives an alkaline cyanide. If iron is used instead of carbon a ferrocyanide is produced, and with sulphur a sulphocyanide. Methyl and ethylic alcohols react on phospham, giving a free secondary amine and the metaphosphate of a primary amine. The reaction of phospham on normal propylic alcohol is more complex, for besides the formation of propylamine we observe a notable quantity of oxide of propyl. With ethylenic glycol the reaction is entirely reversed, and a complete dehydration only is observed; the phospham taking away two molecules of water, forming di-ammonium orthophosphate and setting acetylene at liberty.

Bulletin de la Société d'Encouragement pour l'Industrie Nationale. Series 5, Vol. ii., No. 8, August, 1897.

Report on M. Gossart's Acetylene Lamp.—M. Violle (for the Committee of Economic Arts).—In this lamp the acetylene is generated as is usual by the action of water on carbide of calcium, but its inventor has made use of the action of capillary tubes, whereby the water is brought in contact with the carbide in very minute quan-

ties, and automatically only in the quantity required. By turning off the water tap and the gas outlet tap at the same time the apparatus ceases to work, and no more gas is formed until both taps are turned on again. By turning off the water tap only, the disengagement of gas becomes slower, and gradually stops. If, on the other hand, the gas tap only is turned off, the gas continues to be evolved until the pressure is sufficient to drive back the water in the capillary tubes, which thus act as a safety valve. The production of gas is thus intimately dependent on its consumption.

Journal de Pharmacie et Chimie.
Series 6, vol vi., No. 3.

Decomposition of Iodoform by Light.—G. Fleury.—There is a great diversity of opinion with regard to the action of light on iodoform; the author shows that, as a rule, this decomposing action is of very limited extent, and he thinks that this limit may be attributable to immediate colouring of the liquid to reddish brown by the liberated iodine stopping the violet and ultra-violet rays on the surface, and thus preventing the continuance of the chemical reaction. With the idea of proving the correctness of this theory, some iodoform—dissolved in a mixture of alcohol and ether—was placed in a flask of white glass, in the presence of an excess of finely divided silver. It was then exposed to sunlight, both direct and diffused, and frequently shaken to accelerate the combination of the liberated iodine with the silver. After several days, the liquid no longer became coloured, and on collecting and weighing the mixture of silver and iodide, the amount of iodine resulting from the decomposition was calculated and found to be the exact theoretical amount.

On the Essence of Bitter Fennel.—E. Tardy.—The result of this research shows that the essence of cultivated French bitter fennel contains a dextro-divalent turpentic carbide, a dextro-tetravalent terpene, an inactive carbide called cymen, fenone, estragol, anethol, anisic aldehyd, anisic acetone, anisic acid, and a crystalline body corresponding to the formula $C_{13}H_{14}O_2$.

Estimations of Commercial Albumen.—P. Carles.—Already noticed.

Colorimetric Reaction of Disulphuric Acid.—E. Barral.

Analysis of an Artificial Roasted Coffee.—F. Coreil.—The sample examined was found to be made by moulding a mixture of flour and husk of wheat, potato, leguminous flour, gum, vegetable refuse, &c., and to contain none of the elementary constituents of true coffee berries.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xvii.-xviii., No. 15.

M. Tanret delivered a discourse on the lamented death of M. Schützenberger, paying tribute to his bold and lofty views, aided by a rare talent of observation and experimentation. Thanks to his numerous researches, of which only a small number could be here mentioned, the name of Schützenberger will live for ever in Science, and will find a place among the foremost of modern chemists.

M. Tanret presented a note, on behalf of M. Sabatier, "On Blue Nitrodisulphonic Acid and some of its Salts."

On Exact Cryoscopy. Correction for Surfusation, and a Means of Recognising a Good Cryoscopic Method.—A. Ponsot.—Amongst the methods used for making the correction for surfusation, that of M. Raoult consists of finding experimentally the points of congelation of the solution A' , A'' , corresponding to degrees of surfusation δ' , δ'' , &c.; a graphic curve gives A . The variation for 1° of surfusation from the point of congelation observed in a solution divided by the lowering is called K . He shows that K is variable with the concen-

tration of a solution, and with the nature of the body dissolved.

On a Colorimetric Reaction of Disulphuric Acid.—E. Barral.

On the Thermic Study of Suberic Acid.—G. Massol.—Suberic acid occurs in the form of brilliant white laminated crystals, melting at 139.5° , and slightly soluble in cold water. It forms suberate of potash easily at the ordinary temperature. The neutral salt dried at 100° is anhydrous, and easily dissolves in water. The acid suberate of potash is very little soluble in water; its heat of solution at about 40° is 5.26 calories, that of the neutral salt being 0.92 calories.

On the Thermic Study of Sebacic Acid.—G. Massol.—The acid salts of potash and soda are but slightly soluble in water; the anhydrous neutral sebate of potash dissolves in absorbing 1.33 calories. The heat of formation of neutral suberate of potash is considerable for an acid of so high a molecular weight, viz., 43.99 calories.

General Considerations on the Normal Di-acids of the Oxalic Series.—G. Massol.—The heats of formation of the neutral anhydrous salts of potash for the six acids the author has studied are as follows:— C_2 , oxalic, $=58.97$ cal.; C_3 , malonic, $=48.57$ cal.; C_4 , succinic, $=46.40$ cal.; C_5 , glutaric, $=44.23$ cal.; C_8 , suberic, $=44.76$ cal.; C_{10} , sebacic, $=43.99$ cal.; 2 molecules of acetic acid $=43.72$ cal. He considers that the quantities of heat which measure the chemical affinity decrease as the molecular weights increase, the heat of formation tends to become low, the reciprocal influence of two carboxyls decreases very rapidly by the introduction of one or two CH_2 intermediaries. The influence of two carboxyls ought to betray itself by the possibility of the formation of internal anhydrides. The acid normal alcohols of the lactic series present analogous facts; for instance, the reciprocal influence of acid oxyhydriles and alcohol is shown by the formation of lactones.

On Acetylmethylheptenone.—Ph. Barbier and G. Leser.—Acetylmethylheptenone soda, treated with iodide of ethyl, gives ethylacetylmethylheptenone, boiling at $133-135^\circ$ under 15 m.m. pressure.

Contribution to our Knowledge of Aliphatic Nitramines.—A. P. N. Franchimont.—All the aliphatic nitramines, acid and neutral, when treated with an acetic solution to which is added either α -naphthylamine, aniline, dimethylaniline, &c., give, with a scrap of zinc, either yellow, red, or green colouring-matters. Nitrourea behaves in the same manner. The detailed examination only of one of these colours can throw any light on this reaction.

Boiled Milk.—Experiments undertaken by Dr. Chamouin, first on kittens and afterwards on infants, show that those fed on boiled milk thrive much better than those fed only on milk in its natural state. Not only does the boiling sterilise the milk, but it also renders it much more easy of digestion. The milk must of course be kept covered after it is boiled.—*The Public Health Journal* (New York), August, 1897.

A Rapid and Practical Method for Determining Carbon in Iron.—J. George Heid.—The sample is treated with copper ammonium chloride in the usual manner, and the separated carbon is collected on an asbestos filter, where it is washed successively with water, alcohol, and ether; it is then transferred to a Rose crucible, dried at 120° , and weighed. A stream of oxygen is led into the crucible which is heated over a Bunsen flame, and the carbon is thus burned off in from three to five minutes; the difference in the weight is the "total carbon." The "graphitic carbon" is obtained by dissolving the iron in dilute hydrochloric acid and determining the separated carbon as before.—*Eng. Min. Journ.*, lxiii., 64.

MISCELLANEOUS.

Improvements in the Colorimetric Tests for Copper. — George L. Heath. — Standard ammoniacal copper solutions which are permanent for long periods, may be prepared by replacing nitric acid by sulphuric acid after the solution of the pure copper in the former, provided a considerable excess of ammonia is added and the solution preserved in bottles with stoppers sufficiently tight to prevent any escape of ammonia. In the analysis of lean material for copper, a double precipitation of the iron and alumina by ammonia is found to yield better results, and in less time than either the precipitation by aluminum or a single precipitation by ammonia. — *J. Am. Chem. Soc.*, xix., 24.

International Conference of Leather-Traders' Chemists. — A Conference, as above, holden on Tuesday and Wednesday, Sept. 28th and 29th, at Herold's Institute, Bermondsey (Leathersellers Company's Tanning School), and at which Great Britain, the United States of America, Austria, Denmark, France, Germany, Norway, and Sweden were the countries represented, concluded its proceedings on the 30th ult., by a joint meeting of the leather trade and its allies at Leathersellers' Hall, kindly lent by the Worshipful Company of Leathersellers. The object of the Conference was to arrive at uniformity in the matter of tanning analyses, and formally establish an International Association of Leather-Traders' Chemists, &c. The Conference was opened by Mr. C. T. Millis, Principal of Herold's Institute, and representing the Governors of the Borough Polytechnic, of which the Institute is a Branch; the chair being afterwards taken by Dr. Perkin, F.R.S. The Rt. Hon. W. L. Jackson presided at the Leathersellers' Hall Meeting. As the first President of the International Association the Conference elected Mr. Alfred Seymour-Jones, F.C.S., F.I.C.; Hon. Secretaries, Prof. H. R. Procter (Yorkshire College, Leeds), and Dr. J. Gordon Parker (Herold's Institute, London).

The Cause of Arsenical Poisoning by Wall Papers. — Thomas Bolas, F.C.S. — It has long been recognised that arsenical wall papers do serious mischief, but the work of Gosio and of Emmerling seems to have cleared up that mystery which has surrounded the matter. Certain moulds, including the very common *mucor mucedo*, have a remarkable property of decomposing arsenical compounds with the evolution of volatile products containing arsenic, and the highly poisonous character of volatile arsenical compounds, coming into the system by way of the respiratory organs, is well known. Arsenious acid is, even in small quantities, a highly antiseptic substance, and poisonous to moulds, so the throwing off of the arsenic in a volatile form may be an effort of nature to cast out the poison. The arsenical copper greens, and other colouring-matters containing arsenic, are still used, and, paradoxical as it may appear, it is by no means improbable that the most dangerous wall papers are those containing a mere trace of arsenic, as when the quantity is large the moulds cannot exist. Traces of arsenic may come into wall papers from the imperfect washing of vessels used to contain the more highly arsenical colours. Now that boric acid is very cheap, the old and perhaps forgotten suggestion of Bolley to use a precipitated borate of copper as a green pigment in place of the arsenical green deserves attention. Bolley's green is prepared by taking two parts of blue vitriol (crystallised cupric sulphate) and three parts of borax in separate quantities of cold water and mixing; after which the precipitate is washed and dried. Dyed and printed fabrics now very frequently contain traces of arsenic. — *Journal of the Society of Arts*.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1977.

ON THE COMPOSITION OF CERTAIN CANADIAN VIRGIN SOILS.*

By FRANK T. SHUTT, M.A. F.I.C., F.C.S.,
Chemist, Dominion Experimental Farms.

OF the many investigations carried on by the Chemical Division of the Dominion Experimental Farms during the past ten years, not the least in scientific interest nor in agricultural value have been those which have had for their object the determination of the amounts of plant food in certain typical and virgin soils of the Dominion. The data are not as yet voluminous, for this work is one that consumes much time, and other and more pressing demands have only permitted an intermittent attention to it; nevertheless we have been able to place on record results which go far towards indicating the character of many soils representative of large untilled, or, at all events, but partially settled districts in Canada.

In all, we have submitted to complete analysis about ninety samples. These comprise surface and sub-soils taken from the Atlantic to the Pacific in the various provinces of the Dominion, and, to the best of our knowledge, from areas which had never been manured or cropped.

It is not my purpose to present in this paper all the data obtained, nor to attempt an interpretation of all the figures, chemical and physical, that have resulted from this work, for such would scarcely be possible.† My intention rather is to bring before you the percentage composition of these soils as regards certain of the more important elements of fertility, and to draw such deductions as to relative richness or deficiency in plant food as may seem warranted when comparing the figures with those obtained from the examination of soils in other countries.

The Value of an Ordinary Soil Analysis.

The exact value of a chemical analysis towards ascertaining the fertility of a soil is a question that probably will always be open to discussion, and all present are doubtless aware that no problem in agricultural science has excited more interest or been debated with greater warmth. We are obliged to confess that a knowledge of the amounts of nitrogen, potash, phosphoric acid, &c., as estimated by our present methods of determining "total" or maximum amounts of plant food constituents by strong solvents, is not in itself sufficient for making a diagnosis as to the crop-producing power of a soil. Why this is so will be apparent upon reflection. In the first place, hydrochloric acid of the strength employed in the analysis dissolves from the soil the mineral constituents in much larger amounts than are present in an *immediately* available condition; and, secondly, there are factors other than the amount of plant food present that are equally important in determining a soil's fertility. The physical condition of the soil,—including retentivity of moisture, capillarity, permeability, &c.,—the climatic conditions, including rainfall, mean temperature, sunshine, &c., must all be carefully considered in conjunction with the analytical figures when endeavouring to interpret the latter with a view of ascertaining a soil's probable crop-producing ability. The case is very similar to that of

water analysis, in which it is universally held that all possible information respecting the source and its environment must be in the possession of the chemist before he can intelligibly and correctly give judgment from his figures upon the quality of the water under examination.

It is often urged that our usual method of soil analysis, using hot, strong, hydrochloric acid as a solvent, only indicates the amounts of plant food that *may* become available, not the amounts that are immediately assimilable. This is true, and it is certainly a serious drawback, but it in no wise makes the results of no value, as some would have us believe. It gives, we may suppose, the maximum amounts of the mineral elements present which under the influence of favourable climatic and mechanical conditions may become useful to crops. It shows decisively deficiencies in any of the plant food constituents, if such exist, and consequently affords valuable information regarding the suitability of the soil for various farm crops, and further indicates the direction in which fertilisation may be economically and profitably carried on. Soils with large stores of plant food, even if such be partially or largely in a locked-up condition, have repeatedly been shown to have a greater agricultural value than those that furnish to the same solvent much less amounts. The probabilities are that, other things being equal, soils of the former class will contain, or at all events under favourable circumstances yield, larger amounts of readily assimilable food than those possessing smaller "totals" or maximums. Soils showing percentages of maximums above the average invariably prove fertile, if climatic influences are favourable. We cannot argue very closely, I admit, but from such an analysis we are able to predict possibilities as to productiveness, provided agencies favourable to the unlocking of soil plant food are present.

Soil Tests for ascertaining Available Plant Food.

Pot or plot experiments are as yet, we confess, the only tests that can infallibly indicate a deficiency in *available* fertilising constituents. Such methods consume much time, are cumbersome, and from their very nature scarcely suited to wide application. What is needed is a laboratory method or methods, in addition to those we now use, which will furnish data in accordance with the results obtained by actual soil trials with crops. This is a question that at present many agricultural chemists are engaged upon, and I venture to hope that ere long the renewed interest in this work will result in satisfactory methods being established, both for available mineral constituents and nitrogen.

Dr. Dyer's Work.

In March, 1894, Dr. Bernard Dyer's work on available plant food in soils appeared. It was the beginning of a new era in soil analysis. Since that date increased attention has been paid to this branch of research, and especially so on this continent. Every year sees new and interesting data, the results of the labours of agricultural chemists of the Experiment Stations of the United States. Dr. Dyer, it will be remembered, showed, among other valuable results, that the root sap and the exudation of rootlets possessed an acidity approximately equivalent to that of a 1 per cent solution of nitric acid. From this he argued that such a solution would have a solvent action on the mineral constituents of the soil similar and equal to that exerted by growing crops. Further, he showed that results obtained by this method were strictly in line with the deductions made from the data of actual field trials. He therefore proposed that this solvent should be used to determine available potash and phosphoric acid in soils. Workers in the United States, members of the Association of Agricultural Chemists, besides using this solvent, have proposed and worked with, during the past few years, other solutions, such as ammonium chloride and calcium chloride. None of these, however, have had the support or corroboration of experiments to show that they were similar or comparable in their action upon the soil to the

* Read before the British Association (Section B), Toronto Meeting, 1897.

† The data here referred to are to be found in the Reports of the Chemical Division of the Experimental Farms, 1888—1896.

solvent action of root exudations. Consequently they do not as yet appeal to agricultural chemists with the same force as the solvent proposed by Dr. Dyer.

Solvents employed.

The solvent used by us in the determination of "total" or maximum percentages of the mineral constituents has been hydrochloric acid, sp. gr. 1.115 (corresponding to 22.86 per cent HCl), 10 grms. of the air-dried soil being digested with 100 c.c. of the acid at the temperature of the water-bath for ten hours.

For the estimation of the "available" potash and phosphoric acid, 1 per cent citric acid solution has been employed, digesting 100 grms. of air-dried soil with 500 c.c. of the solvent for five hours at room temperatures.

Standards of Fertility.

It has been remarked that climate and the physical condition of a soil are potent factors in determining fertility. To this might be added the statement that fertility (*i.e.*, crop-producing power) is a relative quality, depending to a large extent on the crop grown. The ability of plants to forage for and appropriate their food varies greatly, so that what might be an adequate supply of food for one might prove an insufficiency for another. Buckwheat and wheat will very well illustrate this variation in foraging and assimilating ability. For these reasons chiefly—for of course there are others—it is impossible to establish rigid standards as regards the minimum amounts of plant food for all crops that must be present in order that a soil may be classed as economically productive.

It is not impossible, however, using a large number of analyses of soils, the productive power of which soils is approximately known, to deduce percentages or limits in plant food, below which, under ordinary circumstances, soils may be considered as deficient or lacking, and above which they may be considered as well supplied or rich in the essential mineral elements. Professor Hilgard, of the California Experiment Station, the highest authority on American soils, considers that less than 0.09 per cent potash indicates a deficiency in this element, and that the limits of this constituent in good soils range approximately from 0.8 to 0.5 per cent in heavy clays, from 0.45 to 0.30 per cent in medium loams, and from 0.3 to 0.1 per cent in sandy loams. Regarding phosphoric acid, he says that 0.2 per cent is sufficient when associated with a good supply of lime, though it may in certain soils reach or exceed 0.3 per cent. Respecting lime, Hilgard states 0.1 per cent in sandy loams as the lowest limit for good crops, 0.25 per cent in clay loams, and 0.3 per cent in heavy clay soils.

Standards of Fertility in Canadian Virgin Soils.

Our data indicate that good agricultural soils in Canada possess usually between 0.25 and 0.5 per cent potash; less than 0.15 per cent, in our experience, points to the necessity, or at all events to the value, of potassic fertilisers, though with good climatic and soil conditions the limit might be reduced to that suggested by Hilgard.

The phosphoric acid in Canadian virgin soils of average fertility lies usually between 0.15 and 0.25 per cent. Some good soils contain from 0.25 to 0.3 per cent, and a few exceed the latter figure. The adequacy, or otherwise, of phosphoric acid in a soil would appear to depend largely on the accompanying amount of lime. Increased crop production has usually followed the application of phosphatic fertilisers to soils containing less than 0.15 per cent phosphoric acid.

Lime ranks next in importance to potash and phosphoric acid in a consideration of the mineral constituents of plant food. I am led to the conclusion that clay soils containing less than 0.1 per cent will have their productiveness increased by a dressing of lime in one or other of its agricultural forms. Peaty soils, and soils generally that are exceedingly rich in organic matter, are

frequently poor in this element. All such have been found to respond to an application of lime, and more particularly so when given in conjunction with potash and phosphoric acid. For these classes of soils, therefore, I deem it advantageous that they should contain at least 1 per cent of lime.

Richness in nitrogen may be measured to a large degree by the organic matter or humus content, though the condition or stage of decomposition of this organic matter is an important factor in determining nitrogen's availability. A large number of our good soils contain between 0.1 and 0.2 per cent, though many reach 5 per cent, and some exceed 1 per cent nitrogen.

In the following brief review of Canadian virgin soils I have not given any detailed data of their physical condition or composition, for the determinations in our laboratory have been confined simply to the separation of the mineral components into (a) clay and fine sand, and (b) coarse sand, according to the method of Schloesing. The results of this separation, together with remarks on the physical condition or tilth of the soils, have been indicated in general terms in discussing the samples. If it had been possible to have made a more extended physical examination I believe the data would have proved most valuable, for the degree of permeability of water and air, the relative size of the soil particles, compactness, water-holding capacity, &c., are important factors towards establishing a soil's suitability for the various agricultural crops.

(To be continued).

ANALYSIS OF SOME PRE-CARBONIFEROUS COALS.*

By W. HODGSON ELLIS.

THE occurrence of anthracite in the calciferous sand-rock of New York, near the base of the Lower Silurian, was recorded by Vanuxem in 1842 ("Geology of New York," iii., 33). It was found in cavities in the rock associated with quartz crystals, sometimes existing within the crystals. He stated that it contained "86½ per cent of carbon; 2 of light cream-coloured ashes, which were of silex; and 11½ per cent of water," *i.e.*, volatile matter. He attributed its origin to the action of heat on bituminous matter contained in the rock.

Sterry Hunt subsequently described ("Geology of Canada," 1863, p. 524) a substance of like nature filling veins and fissures in rocks of similar age near Quebec and on the north shore of Lake Superior.

E. J. Chapman (*Canadian Journal*, vol. x., p. 410) described an anthracite from Lake Superior, occurring in a banded vein with quartz and iron pyrites, an analysis of which gave—

Moisture	..	..	2.08
Volatile matter	..	..	3.56
Fixed carbon	..	..	94.36
Ash	..	..	0.00
100.00			

In his "Minerals and Geology of Central Canada," p. 143, Chapman proposed the name "anthraxolite" for this pre-carboniferous anthracite, chiefly because its mode of occurrence differs from that of the anthracite of the coal measures.

In *Bulletin No. 2* of the Ontario Bureau of Mines, November, 1896, the discovery is reported of a remarkable deposit of coaly material occurring as an irregular vein, about 6 feet thick, in black fissile seats of Cambrian age, situated near Sudbury, the locality of the famous

* Read before the British Association (Section B), Toronto Meeting, 1897.

Canadian nickel mines. The discovery roused great public interest, and hopes were entertained that the coal would prove of commercial value. Specimens were sent to Dr. Dawson and Mr. Hoffmann, of the Geological Survey of Canada, who reported it as consisting of anthraxolite and quartz, the latter constituting 55·95 per cent of the whole. Dr. A. P. Coleman was sent by the Ontario Government to examine the deposit, and he described it as a lustrous black mineral, forming "small plates or irregular cubical blocks, the largest observed being three-quarters of an inch square. Between the plates or cubes there is generally more or less quartz, and in some weathered portions on the surface the quartz remains as a porous cellular mass." Dr. Coleman considered that "the source of the anthraxolite is probably bituminous matter contained in the adjoining beds of slate. By metamorphic action most of the volatile matter has been removed from the once fluid or plastic bitumen, leaving the present cracked and quartz-cemented anthraxolite."

In the *Proceedings of the Canadian Institute* for February, 1897, will be found a detailed account of this interesting deposit by Mr. G. R. Mickle, and also a chemical analysis by Mr. W. Lawson and myself.

The pure coaly substance has a specific gravity of 1·865 and a hardness between 3 and 4. It burns with difficulty, giving off a good deal of heat (a sample containing 3·99 per cent ash gave, in Fischer's calorimeter, 7·490 calories per grm.) and leaving a light fawn-coloured ash consisting of silica with a trace of oxide of iron. The quantity of ash varies in different samples, some containing as high as 60 per cent. The average as far as yet examined is about 20 per cent.

The following is the proximate analysis of an average and of a selected sample:—

	Average.	Selected.
Moisture	4·0	4·0
Volatile matter	1·3	1·8
Fixed carbon.. .. .	74·2	90·1
Ash.. .. .	20·5	4·1
	100·0	100·0

The ultimate analysis of a carefully picked specimen, freed from moisture, was as follows:—

Carbon	94·92
Hydrogen	0·52
Nitrogen	1·04
Sulphur.. .. .	0·31
Oxygen	1·69
Ash	1·52
	100·00

The striking point in this analysis is the small percentage of hydrogen, which is less than that given in any published analysis that I have been able to find of anthracite of the carboniferous age.

In this respect, as well as in its physical properties, it bears a striking resemblance to the mineral schungite described by Inostranzeff as occurring in Huronian rocks on the shore of Lake Onega, in Russia. This mineral has a specific gravity of 1·84 and a hardness of 3·5 to 4, and contains 99·2 per cent of carbon and 0·4 per cent of hydrogen. The Sudbury coal contains more oxygen, but otherwise the resemblance between the two is very striking. The so-called graphitoid of Sauer, from the Saxon Erzgebirge, with 99·8 per cent of carbon and 0·2 per cent of hydrogen, is another closely allied mineral.

It seemed of interest to compare the composition of the Sudbury anthraxolite with that of specimens from other Canadian localities, and accordingly Mr. Lawson and myself made an analysis of a specimen from the neighbourhood of Kingston, for which we were indebted to Mr. W. G. Miller. It occurs in a vein of barite cutting Lower Silurian limestones. The anthraxolite coats and fills

crevices in the barite. The mineral is duller and softer than the Sudbury anthraxolite. Its specific gravity is 1·365. The analysis of the dry substance gave the following figures:—

Carbon	90·25
Hydrogen	4·16
Nitrogen	0·52
Sulphur.. .. .	0·66
Oxygen	3·69
Ash	0·72
	100·00

Since then, through the kindness of Dr. G. M. Dawson, Director of the Geological Survey of Canada, I have had the opportunity of examining three other specimens of anthraxolite—one from Cap Rouge, near Quebec, and two from the peninsula of Labrador.

Anthraxolite from Cap Rouge.—A black coaly substance occurring in cracks of "beds of a hard jaspery character," associated with black bituminous shales of Lower Silurian age ("Geology of Canada," 1863, p. 203).

The analysis of this specimen was as follows:—

Moisture	0·19
Ash	9·02
Carbon	82·90
Hydrogen	5·50
Oxygen	2·37
	100·00

(Including nitrogen and sulphur not determined).

Anthraxolite from Lake Mistassini.—A bituminous mineral, with the lustre and colour of anthracite, filling cavities in calcite veins in limestones of Cambrian age (A. P. Lowe, "Report of the Geological Survey of Canada," 1895, 267 L.). I found the composition of this specimen to be—

Moisture	1·75
Ash	1·07
Carbon	92·71
Hydrogen	1·02
Oxygen, &c.. .. .	3·45
	100·00

Anthraxolite from Lake Petitsikapau, District of Ungava, Labrador Peninsula.—In veins, with quartz in Cambrian shales. Foliated with plates at right-angles to the walls. Collected by Mr. A. P. Low. A purer specimen gave Mr. Hoffmann ("Report of the Geol. Survey of Canada," 1894, 66 R.) the following results on proximate analysis:—

Water	3·56
Volatile matter	2·48
Fixed carbon	86·83
Ash	7·13
	100·00

My specimen had the following composition:—

Moisture	0·81
Ash	48·37
Carbon	49·39
Hydrogen	0·67
Oxygen, &c.. .. .	0·76
	100·00

It will be seen that in none of these is the hydrogen so low as in the Sudbury anthraxolite. It will also be seen that they vary among themselves as much as do the varieties of coal which occur in the beds of the carboniferous period. This variability is in strict accordance

with the view that they result from the metamorphosis of bitumen, of the various stages of which metamorphosis they serve as excellent illustrations. To make this more striking, I have calculated the analysis to the dry ash free substance, and for the sake of comparison I have added the analyses of petroleum, asphalt, and albertite—the latter a coal-like mineral occurring in a vein in rocks of lower-carboniferous age in Nova Scotia, for some time successfully worked as fuel. At the other end of the scale I have given the analyses of schungite and graphitoid.

	Locality.	Carbon.	Hydro- gen.	Oxygen, &c.
1. Petroleum ..	Pennsylvania ..	82.0	14.8	3.2
2. Asphalt ..	Mexico ..	83.3	10.8	5.9
3. Albertite, ..	Nova Scotia ..	86.0	9.0	5.0
Anthraxolite .	Kingston ..	90.5	4.2	5.5
"	Cap Rouge. ..	91.3	6.2	2.5
"	Lake Mistassini	95.2	1.2	3.6
"	Sudbury ..	96.4	0.5	3.1
"	Lake Petitsikapau	97.1	1.3	1.6
4. Schungite ..	Lake Onega ..	99.2	0.4	0.4
5. Graphitoid ..	Erzgebirge. ..	99.8	0.2	0.0

1. Ste.-Claire Deville, *Comptes Rendus*, lxvi., 442.
2. H. Endemann, *Journ. Soc. Chim. Ind.*, xv., 872.
3. Wetherill, *Trans. Am. Phil. Soc. Philad.*, 353, 1852.
4. Inostranzeff, "Neues Jahrbuch für Mineralogie," i., 97, 1880; i., 92, 1886.
5. Sauer, *Zeit. Geol. Gesell.*, xxxvii., 441, 1885.

From these latter to graphite, the transition is rather one of crystalline form than of chemical composition, as indicated by analysis.

Chemical Laboratory,
School of Practical Science, Toronto,
August 18, 1897.

THE CARBOHYDRATES OF THE CEREAL STRAWS.*

THE work upon the barley crop of 1896, which was reported in outline to the Chemical Section in a paper read by Mr. Cross, has been more fully dealt with in a paper read subsequently, and published in the *Journal of the Chemical Society*, 1896, pp. 804–818. The subject was also dealt with from the more special point of view of the relation of the furfuroic constituents of these straws to the important problems of animal digestion and alcoholic fermentation in a paper published in the *Journal of the Fed. Inst. of Brewing*, 1897, Pt. 1.

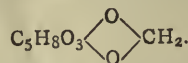
The investigations have been continued without interruption. We have further and more closely studied the products of acid hydrolysis of the cereal straws and of the celluloses isolated from them, and the main results of these researches are embodied in a paper read at the meeting of the Chemical Society, London, on June 17.

Generally the results of the preceding paper (*loc. cit.*) are amplified and confirmed. As it had been previously shown that the furfural-yielding constituents of fodder plants are in large measure hydrolysed and assimilated by the animal organism, so the evidence is accumulating that certain of these compounds when fully hydrolysed (to monoses) by artificial processes are susceptible of alcoholic fermentation.

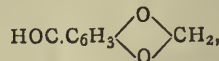
It having been finally established that the pentoses themselves are entirely resistant to the attack of the yeast cell, it follows that we are dealing with a class of furfural-yielding carbohydrates, *not* pentoses.

At the present time the reactions of these compounds

clearly indicates that they are pentose-derivatives, and most probably methylene ethers of the C₅ sugars of the general formula—



It is difficult to devise reactions of decomposition or synthesis by which such a constitutional formula could be finally verified. The literature of the analogous compound dipironal—



but with an aromatic in place of a pentose residue, may be cited in evidence of the exceptional difficulty of the problem presented.

The authors are glad to report that through the kindness of friends they have now access to a vessel enabling them to operate upon a large weight (7 kilos.) of the raw materials.

Working upon this extended scale, and upon the basis of the results established by long investigation and previously reported to the Section, we may confidently expect more positive and, we hope, final results.

ANALYSIS OF A BLACK SILK DRESS.

By Dr. T. L. PHIPSON,
formerly of the University of Brussels and the Laboratoire de Chimie
Pratique, Paris.

A LADY paid a visit to my laboratory a short time ago and enquired whether I had ever made an analysis of a silk dress. I confessed that among the many hundreds of various things examined in the course of a long series of years, I had only been called upon hitherto to discover the nature of certain poisonous colours applied to silk gloves and silk stockings, but I could find no analysis of a silk dress on my books.

Nevertheless, my fair visitor was anxious that I would undertake this analysis for her, and also expressed the desire that I would publish the results in the *CHEMICAL NEWS*, a journal which, she said, she was in the habit of reading.

Her husband was interested in the silk trade, but had little knowledge of chemistry, whilst she herself had gone through a course of practical science, and wished to convince him that the value of silk material might be ascertained by analysis. She explained, moreover, that the material she placed in my hands was intended for a silk blouse, that it was a medium quality of silk (neither the most expensive nor the cheapest), and she had heard that certain black silks, when stored in large bulk, in dry hot weather, were liable to spontaneous combustion, that two such cases were already known (one in Paris and one in New York), and she was therefore anxious that her husband should increase his insurance upon his stock of silk goods.

The following days I devoted myself to the work in question, and I found that the material contained a very large amount of substance that was not silk at all; in fact, that it was considerably "weighted," as all silks are to a greater or less extent. It would not burn with flame, but smouldered away like tinder and left a large amount of ash, the principal ingredient of which was oxide of tin. Indeed, I have examined specimens of poor tin ore from Cornwall that did not contain more tin than this material for a lady's blouse; and I at once realised the fact that the silk dresses worn by the ladies we see daily parading in Regent Street and Bond Street, taken together, would represent a Cornish mine of very fair quality.

But this enquiry has brought to light a new and powerful application of chemical analysis—already one of

* Report of the Committee, consisting of Professor R. Warington (Chairman), C. F. Cross (Secretary), and Manning Prentice. (Drawn up by the Secretary). Read before the British Association (Section B), Toronto Meeting, 1897.

the greatest powers in the hands of man—namely, that the duration or wear of a silk dress can be determined by analysis. It used to be said that a garment of pure silk would last a life-time; but I happen to be acquainted with a very clever young milliner who has assured me that the silk material I have examined, if worn every day, would not last more than three months—meaning, of course, that by that time it would be "utterly shabby, greasy-looking, and showing the threads."

The figures obtained in my analysis are as follow:—

Water	11'43
Ash (mostly tin oxide and silica)	14'30
Real silk	28'14
Organic matters, &c., not silk	46'13
	100'00
Nitrogen	4'76

The weighting of silk is now carried on to so great an extent in France, Germany, and Switzerland, that some foreign silks get shabby with a few weeks' wear; and we are seriously told that the public prefer these cheap products, as the fashions of jackets, blouses, and skirts change so rapidly that it would be useless to purchase silk of better quality!

Casa Mia, Putney, S.W.

A MODIFICATION OF THE CYANIDE TITRATIONS OF COPPER.

By HARRY BREARLEY.

THIS paper is offered with some diffidence; its subject-matter has been so often examined and discussed by those daily interested in its accuracy. A belief that the modification is also an improvement, and its general bearing on a subsequent paper must stand as an apology for any seeming presumption on the part of one only rarely engaged with copper estimations.

For each element in the "alkaline acetate" separations the question—"How shall the separated element be estimated?" had in all cases to be primarily settled. For obvious reasons, when a large number of estimations had to be made a volumetric process was preferred.

The cyanide method of Parkes and the iodide method of De Haen are, of all volumetric methods for copper, perhaps the most widely known and practised. Both, however, are at a disadvantage when the copper percentage is low and the bulk of the solution also large. This being so, the proposed acetate separations of iron and copper would entail an additional operation before the estimation could be made, and thus destroy for the particular purpose a volumetric method's chief charm—rapidity.

The latter stages of the cyanide titration are rather indistinct. The solution assumes a pale violet or lavender tint, which fades very gradually independently of any cyanide additions; and so it becomes not altogether impossible, in following the changes—unconsciously, of course—to have one's expectations warping one's judgment. The suggestion to titrate only to a pale lavender tint (Clowes and Coleman, "Quant. Anal.," 2nd edition, p. 195) does not greatly amend this uncertainty.

The proposed modification lies in the application of the silver iodide indicator to the usual titration.

If the silver iodide be formed in the solution before the cyanide is added it is no indicator whatever; because the suspended iodide is equally or more active to the cyanide than is the ammoniacal copper, and so the solution clears sometime before the blue colour is discharged. This difficulty is obviated by adding cyanide to the usual point, or thereabouts, and then adding potassium iodide

and going back to a permanent turbidity with silver nitrate. As a matter of fact, the turbidity is not really permanent, but only persistent; because, on standing, any turbidity would disappear on account of its greater affinity for the cyanide, and a corresponding amount of copper being displaced would reproduce the characteristic blue colouration.* By this means, the end-reaction becomes certain, and with identical liquids gives good results. In the presence of additional compounds it exhibits its usual waywardness.

In 1888, Mr. J. L. Davies (CHEMICAL NEWS, lviii., 131) very modestly suggests the substitution of soda carbonate for ammonia. The carbonate is to be added in such excess as to partly dissolve the precipitated copper, and then the usual end-reaction is relied on. Mr. Merry modifies the original idea by adding enough tartaric acid to re-dissolve the precipitated carbonate to a clear blue solution, and then titrating. Eighteen months later (CHEMICAL NEWS, lxi., 183), Mr. Fessenden suggests a like change, especially stipulating that the soda carbonate be added to a solution containing free nitric acid, so as to completely effect the re-solution of the precipitated copper.

It was claimed for this modification that the final colouration did not lag, as with ammonia, and that the values are not so greatly affected by additional alkali or alkaline salts.

These suggestions occupy only two short letters to the editor; and, judging from the persistent way in which the ammonium titration is still practised and recommended, seem to have been undeservedly neglected.

A mere comparison of similar solutions made alkaline with ammonia and soda carbonate respectively, shows the latter under unfavourable conditions. But an addition of cyanide to the latter both changes and deepens the tint, and this deepening keeps pace with the progressing titration for some time. It cannot even be pretended that the colour vanishes like phenolphthalein in alkalimetry, but it is no exaggeration to say that its sensitiveness is two or three times greater than when ammonia is used.

There are arranged below some titrations with soda carbonate and with ammonia under varying circumstances. Attention is chiefly paid to the former on account of its evident superiority.

The iodide indicator cannot be added before the titration with the soda any more than with the ammonia solution, although the turbidity holds out much longer in the former case; and the copper having once combined with the cyanide is not decomposed by the silver iodide turbidity.

It should be noticed that if cyanide be added to an alkaline solution of copper until the colour is just discharged, the whole of the cyanide has not combined with the copper. For instance, a solution containing 0.03 gm. of copper, if titrated as usual, would contain as "surplus" cyanide about 10 per cent of the total quantity used. Of course, by prolonged titration this surplus could be materially decreased, but—within reasonable time—it cannot be eliminated altogether.

For the following tests the cyanide is added until the colour is discharged, or very nearly so, and the solution allowed to stand five or ten minutes. Prolonged standing very slightly increases the amount of cyanide used, or more intelligibly perhaps, decreases the surplus. The bulk of solution throughout is 250 c.c., the amount of copper 0.02 or 0.03 gm.

* The following remarks by Field (CHEMICAL NEWS, i., 26) are interesting:—"When a soluble cyanide is added to a solution of a salt of copper, in a considerable excess of ammonia, until the liquid is perfectly colourless, the addition of nitrate of silver restores the deep blue colour, and an argento-cyanide of ammonia or of ammonia and copper is formed. There are a great variety of compounds consisting of silver, copper, cyanogen, and ammonium; one especially crystallises in splendid dark blue octahedra, and contains more than 60 per cent of silver. Another compound separates in pearly scales and contains a larger proportion of copper."

Influence of Alkali.

	5	10	15	20 c.c.
Excess of ammonia ..	5	10	15	20 c.c.
Nett KCN	27.7	27.35	26.55	26.12 "
Excess of soda carbonate ..		10	20	40 "
Nett KCN		20.42	20.65	20.75 "

The strength of the alkalis were—Ammonia, 0.880, diluted to four times its bulk; soda carbonate, 200 grms. per litre. The results in the ammonia series seem to be at variance with common experience; they are not really so. The amount of cyanide needed to discharge the colour did increase with the volume of the ammonia, but in a greater degree the needful surplus cyanide also increased, and hence the reversed order of progression.

With an excess of only 5 c.c. of dilute ammonia, a final turbidity equivalent to one or two tenths of a m.grm. of copper will persist for several hours. As the ammonia increases in amount this time would decrease until, with an excess of 40 or 50 c.c., it becomes impossible owing to the rapid interchange of silver and copper, to observe the disappearance of the last traces of free cyanide other than by the returning blue colouration.

Influence of Volume.

Volume titrated ..	100	200	300 c.c.	
Soda	21.16*	20.42	20.01	" KCN nett.
Ammonia	19.13	18.05	17.37	" " "

When the sample marked with an asterisk was made up to about 300 c.c. with water, the final turbidity disappeared, and to reproduce it required silver nitrate equivalent to 1.02 c.c. KCN, which subtracted from 21.16 brings the value into line with the "300 c.c." sample. The corresponding ammonia sample behaved similarly, but not with so great accuracy.

Proportion of Copper.—The volume of the solution was constant, 200 c.c.

	0.005	0.01	0.02	0.03 grm.
Copper	0.005	0.01	0.02	0.03 grm.
Soda	4.85	10.1	20.4	30.67 c.c. KCN
Ammonia	—	8.8	18.05	27.5 " "

Excess of Cyanide.—These tests are intended to show the influence of adding more or less cyanide than a proper amount. This irregularity might happen either inadvertently or otherwise.

	I.	II.	III.	IV.
Total cyanide added	25.5	30.5	37.0	40 c.c.
Nett cyanide	21.6	25.95	28.55	28.95 "

This is an ammonia series. Test I. was decidedly, and test II. very faintly, coloured on adding the silver nitrate. The very considerable variation in the nett cyanide make the proposed iodide indicator of lessened value; because any error in noting the colour indication—the uncertainty of which provides the only excuse for using the AgI—would be only partially corrected by the added silver salt. The soda titration under like conditions is almost free from this error. Thus—

Total KCN	25.1	27.0	30 c.c.
Nett	20.5	20.6	20.78 "

This degree of constancy, taken in conjunction with the increased delicacy of the decolourisation, makes the end-reaction of a soda titration as delicate as the silver iodide is known to be under the most favourable circumstances. It is indifferent also to the soda titration in what manner the cyanide is added. Thus, when the required amount was run in quickly and then shaken, the net result was 20.38 c.c. KCN; when added in drops, shaking meanwhile, the result was 20.33 c.c. KCN

Alkali Chloride.

Ordinary HCl as Na(Am)Cl. C.c.

	0	5	10	20
Soda	20.4	20.4	20.52	20.52 c.c. KCN
Ammonia	28.41	31.4	32.65	33.3 " "

Alkali Nitrate.

HNO₃ (1.20) as Na(Am)NO₃. C.c.

	0	5	10	20
Soda	20.4	20.38	20.43	20.61 c.c. KCN
Ammonia	18.05	19.62	20.32	— " "

With the larger amounts of soda chloride and nitrate the final turbidity is, in the former case very slightly, and in the latter unmistakably, ill-defined. Only the nitrate deserves more than a passing notice.

The whole of the variations at present under consideration were made on a solution containing an exaggerated indicator, but no copper. They were all without appreciable influence, except soda nitrate. The nature of the difficulty is set forth in the table.

HNO₃ (1.20) as NaNO₃. C.c.

	5 c.c.	5	10	20
Excess soda carbonate {	5 c.c.	6.3	7.27	7.5
	10 "	—	6.37	7.12
	20 "	—	6.35	6.35

The values are c.c. KCN. The table also points to the remedy, namely, an increasing excess of soda carbonate with increasing quantities of soda nitrate. Any error due to this irregularity could be detected by adding more soda carbonate. In case there was a previous insufficiency, the turbidity would clear, and then titration would finish in the usual way. A proper titration is not appreciably affected by the further addition of carbonate.

Alkali Acetate.

33 per cent acetic acid as acetate. C.c.

	0	5	10
Ammonia	29.1	31.2	32.1 c.c. KCN

Grms. crystal. soda acetate.

	0	2½	5	10
Soda	20.35	20.4	20.63	20.63

The most casual comparison of the two series of results greatly favours the soda cyanide process. R. T. Thomson, in a paper on "The Interference of certain Metals with the Accuracy of the Cyanide Estimation of Copper" (CHEMICAL NEWS, xxxiii., 152), shows that in ammoniacal solutions the salts of sodium and potassium have no appreciable effect. J. J. and C. Beringer (CHEMICAL NEWS, xlviii., 111, reprint from the *Proceedings of the Cornwall and Devon Miners' Association*) come nearer still to discovering the advantage of complete soda titrations. They say:—"It seemed to us that the interference of the ammonia salts is due to a reaction with potassic cyanide by which ammonium cyanide is formed, and that the excess of cyanide is required either because ammonium cyanide decomposes more rapidly or because it is less active than the corresponding potassic salt. In this case, the use of soda or potash for neutralising the excess of acid used for dissolving the ore would eliminate the disturbing element." And subsequently they show that the sulphates, nitrates, and chlorides of potash and soda have little or no effect on the cyanide titration; but the advisability of avoiding ammonia altogether does not seem to have suggested itself to them.

It has been previously noticed that Mr. Davies performs the titration without completely re-dissolving the precipitated carbonate. Mr. Merry effects the complete solution with tartaric, and Mr. Fessenden with nitric acid. So far as the colour reaction is concerned, it may be perfectly indifferent which acid is used. But if the iodide end-reaction be adopted, it is no longer an indifferent matter. One objectionable feature of nitrates has been already pointed out. There is another; and, if nitric acid is used alone, a more unpleasant one.

If four vessels containing solutions of copper acidified respectively with nitric, acetic, sulphuric, and hydro-

chloric acid are treated with an excess of soda carbonate, each will hold a clear solution. Let the solution be titrated with cyanide, and then, on the clear and colourless solution, after adding potassic iodide, carefully superstratify* dilute silver nitrate. In the first solution (nitric) the silver iodide will lose its lemon colour and become a dirty brown. The same change will take place decreasingly in the acetate and sulphate solutions. When these turbidities are dissolved by shaking they produce a more or less inky tinted solution, and make the final turbidity less easily recognisable. Hydrochloric acid is perfectly free from this defect.

In case the salts producing—or, perhaps better, allowing—this dirty colouration to be produced (because it is not noticeable except in presence of copper), are unavoidably present, the addition of soda chloride will ensure a clean turbidity. The inky tint is not permanent, but it disappears very slowly.

Interference of certain Metals.

The interference of nickel, cobalt, zinc, mercury, silver, and—less noticeable because of rarer occurrence—gold, platinum, and palladium, is known to be of such extent as to invalidate the cyanide titration of copper. Indeed nickel, cobalt, mercury, and silver may be quantitatively estimated by these means. There are, however, a large number of compounds whose effect it would be well to study anew under conditions which are altogether independent of colour reaction. The presence of those compounds which form a precipitate either before or during the titration are especially noteworthy in this connection.

Only the following three metals have been already experimented with. In each case 0.05 grm. of copper was present.

Iron.—Added as Fe_2Cl_6 .

0	0.01	0.03	0.05 grm. Fe
50.3	50.4	50.35	50.0 c.c. KCN
0.0500	0.0501	0.0500	0.0496 grm. Cu
—	0.0001	0.0000	0.0004 Error.

It is quite impossible under these conditions to make any use of colour reaction. The precipitate remains suspended for some time, and is so finely divided as to pass through a moderately porous paper. An expansive asbestos filter gives a clear filtrate.

Aluminium.—Added as Al_2Cl_6 .

0	0.1	0.03	0.05 grm. Al
50.3	50.6	51.1	51.1 c.c. KCN
0.0500	0.0503	0.0508	0.0508 grm. Cu
—	0.0003	0.0008	0.0008 Error.

The colour reaction can be distinctly followed. The variations accord with ammon. cyan. titration when performed as usual.

Manganese.—Added as MnCl_2 .

0	0.01	0.03	0.05 grm. Mn
50.3	47.35	47.3	47.4 c.c. KCN
0.0500	0.0471	0.0470	0.0471 grm. copper
—	0.0029	0.0030	0.0029 Error.

The colour change is no guide. The results are in accord with those by the usual titration.

Considerable attention has been paid to the cause of these low values, though without, so far as I know, any satisfactory explanation being come to. The deficiency here is somewhat less than the usual amount. Thus Field (CHEMICAL NEWS, i., 63) finds a deficiency of 12 per cent, Thomson a deficiency of 20 per cent.

It is noteworthy that the deficiency seems to be inde-

pendent of the amount of manganese present. No especial emphasis is laid on this point. The tests were arranged merely to decide whether or no the manganese would act as usual, and, although interesting in the above character so far as they go, they are altogether inadequate to support a far-reaching pronouncement.

A more extended list of added metals will be given later, together with some means of obviating the interference and applying the corrected process to the determination of copper in copper alloys.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING AUGUST 31ST, 1897.

By SIR WILLIAM CROOKES, F.R.S.,
and

PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, *Metropolis Water Act, 1871.*

London, September 10th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 175 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from August 1st to August 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in a previous report.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 175 samples examined during the month all were found to be clear, bright, and well filtered.

The rainfall at Oxford during August was 3.94 inches, and was very fairly distributed, with the exception of 0.88 inch which fell on the 30th; the average fall for the last 30 years during this month is 2.32 inches; we have therefore had an excess of 1.62 inches. This figure is also the actual excess for this year, to the end of August.

Our bacteriological examination of 235 samples taken by us have given the following results; we have also examined 41 other samples, from special wells, stand-pipes, &c., making a total of 276 in all:—

	Microbes per c.c.
Thames water, unfiltered (mean of 25 samples)	3420
Thames water, from the clear water wells of five Thames-derived supplies (mean of 117 samples)	51
Ditto ditto highest	775
Ditto ditto lowest	10
New River, unfiltered (mean of 25 samples)	3420
New River, filtered (mean of 23 samples)	44
River Lea, unfiltered (mean of 25 samples)	5846
River Lea, from the clear water well of the East London Water Company (mean of 21 samples)	64

The fact that, in spite of the great excess of rain, the number of microbes per c.c. has decreased in the case of every Water Company's supply, except one, which remains the same as during the previous month, shows

* I have elsewhere pointed out that the rapidity with which the resulting turbidity disappears indicates the approaching end-reaction.

that the filtering appliances of the companies are in excellent working order.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.
JAMES DEWAR.

THE ACTION OF NITRIC ACID ON TRIPHENYLMETHANE.

By E. S. SMITH.

THE method of preparing trinitrotriphenylmethane by the action of fuming nitric acid on triphenylmethane is described by Fischer (*Ann. Chem., Liebig*, xciv., 254). In an attempt to make this preparation I departed from the exact directions of Fischer, and obtained a substance shown by analysis to be triphenylcarbinol, $C_6H_5(C_6H_4)_3COH$. Following are the details of the work:—I placed in a flask an unweighed quantity of triphenylmethane recently crystallised from benzene, and probably still retaining a little benzene; poured in an unmeasured quantity of nitric acid (sp. gr. 1.34), and then some fuming nitric acid; allowed the mixture to stand a short time, then heated on the sand-bath for a few minutes. After the reaction was ended a large amount of water was poured in. The reddish yellow precipitate formed was filtered off on a platinum cone. By pressing down the precipitate a small amount of a red oil, having the smell of nitrobenzene, was forced out. The solid substance was purified by treating it with hot glacial acetic acid, crystallising it from a mixture of benzene and glacial acetic acid, re-crystallising from benzene, and finally from alcohol. The purified substance is white, resembles triphenylcarbinol, and melts sharply at 161° (uncorr.). A test for nitrogen by the Prussian-blue method showed absence of nitrogen.

0.138 grm. gave 0.0773 grm. water, and 0.4458 grm. carbon dioxide, indicating the following composition:—

	Calculated for triphenylcarbinol.	Found.
C ₁₉	87.80	88.10
H ₁₆	6.15	6.22
O	6.15	—
	100.00	

Several attempts to confirm this work by repeating it were, on the whole, unsuccessful, though various modifications in the strength of acid, manner of heating, &c., were tried. In one case a very small quantity of a white crystalline substance was obtained, which, after re-crystallising from benzene, melted at 161° .

Nitric acid of sp. gr. 1.42 does not act upon triphenylmethane until the temperature is raised to about 100° ; but the product obtained in this case, as in most of the other experiments, was a waxy substance of salmon colour, from which I was not able to get a crystalline compound.

I could find no record of triphenylcarbinol having been made in this manner; the usual method of preparation is to oxidise the triphenylmethane with chromic acid.—*American Chemical Journal*, xix., 702.

Condy's Fluid.—Notice of Removal.—The Proprietors of "Condy's Fluid" notify that they find it necessary to remove to more extensive and commodious premises. All communications should in future be addressed to Condy's Fluid Works, 65, Goswell Road, London, E.C.

PROCEEDINGS OF SOCIETIES.

THE CHEMICAL AND METALLURGICAL SOCIETY OF SOUTH AFRICA.

Meeting held on July 17, 1897, at Johannesburg.

Mr. CHAS. BUTTERS, President, in the Chair.

THE PRESIDENT delivered his Inaugural Address, in which he first referred to the history of chemistry and metallurgy on the Johannesburg gold-fields. Until now there has been but one industry, viz., gold-mining. Properly speaking there is no Chemical Industry, as, whatever chemical processes there may be at work, all have to do intimately with the extraction of gold. Silver and copper are both at the Willows and the Albert silver-mines, and silver-lead at the Transvaal silver-mines; these mines, however, have not, owing to the excessive cost of production, been yet made to pay. An attempt is being made to establish the manufacture of sulphuric acid, but the only factory yet at work produces acid of low commercial strength only. This is an important matter, as it will largely influence the production of cheap and trustworthy dynamite. Glass is being manufactured successfully at Pretoria. The coal in the Transvaal may be considered as practically inexhaustible, but the percentage of ash is extremely high, viz., 20 to 25 per cent, and the quality is very variable. Galena is very plentiful in the Transvaal, going as high as 80 per cent of lead; the smelting of the ore is done locally, but the demand is limited. But, as was pointed out above, the one industry that can, to a certain extent, survive the repressive measures of the authorities, is gold-mining, and it is to gold only that the country can at present look for commercial prosperity. A great deal of money and thought is now being expended on recovering the last traces of the precious metal which only a few years ago was allowed to run to waste; tailings have been regularly treated, and now managers are turning more attention to slimes.

Mr. D. J. WILLIAMS's paper on "Some Notes of the Estimation of Lead in Slags" was next discussed, after which followed the discussion of Mr. E. H. JOHNSON's paper "On the Reduction of Zinc-Gold Slimes."

Mr. CALDECOTT mentioned that he had used acid sulphate of soda or potash instead of sulphuric acid for this operation; he found it cheaper and better.

Mr. McBRIDE, after paying tribute to the excellence of Mr. Johnson's paper, could not agree with him as to the increased cost of the acid process; he found three-quarters of a pound of acid enough for one pound of moist zinc-gold chips. He then gave details of his own system of cleaning up, which is so intimately connected with the acid treatment as to be almost inseparable from it in a discussion of this kind.

Mr. CROSSE had noticed that some bars assayed as high as 825 fine, while others went very much lower; he therefore analysed some, and found iron, nickel, cobalt, and lead, the three latter not being removable by sulphuric acid.

Mr. J. R. WILLIAMS then read a paper "On the Treatment of Battery Slimes." It is four years since the author first commenced experimenting on the recovery of gold from battery slimes, and he has now succeeded in evolving a process which works in a most satisfactory manner. Briefly it is as follows:—After separating the tailings, the slime water flows through a launder, where enough lime (in the form of milk of lime) is added to precipitate the slimes in a flocculent form; regularity of feeding the lime plays an important part in this process; an excess is as bad as too little. After mixing, the liquid passes through a series of large settling pits, 10 feet deep, from whence the water runs away practically clear. The sediment, with about 10 per cent of water, is then

pumped up, and treated with a 0.61 per cent solution of cyanide, and the gold recovered in the usual manner. The profit made during the months of April and May on this process alone was equal to a 15 per cent per annum dividend on the capital of the Company (Crown Reef G. M. Co.). The discussion of the paper was postponed.

Mr. W. A. CALDECOTT then read the following paper: "*The Solution of Gold in Accumulated and other Slimes.*" When battery slimes are settled in dams or pits, certain reactions take place which result in the formation of decomposition products, among which is ferrous sulphide, FeS , derived from the decomposition of pyrites, FeS_2 . This compound imparts a dark grey or black colour to the slimes where it occurs in any quantity, and is easily identified by the smell of sulphuretted hydrogen given off on the addition of acid. The exact stages of the decomposition may be open to discussion, but the author inclines to the opinion that the main stages of increasing oxidation are as follows:—

1. FeS_2 Iron pyrites.
2. $\text{FeS} + \text{S}$ Ferrous sulphide and sulphur.
3. $\text{FeSO}_4 + \text{H}_2\text{SO}_4$.. Ferrous sulphate and sulphuric acid.
4. $\text{Fe}_2(\text{SO}_4)_3$ Ferric sulphate.
5. $2\text{Fe}_2\text{O}_3 \cdot \text{SO}_3$.. Insoluble, basic, ferric sulphate.
6. Fe_2O_3 Ferric oxide.

During the treatment of slimes by cyanide, the presence of finely divided ferrous sulphide causes abstraction of oxygen from the solutions, whereby the solution of gold is prevented. Other ferrous compounds, such as ferrous hydrate, react in the same manner, as does also the decomposing organic matter always present in slimes. The obvious way to prevent this interference is to supply oxygen, either by pumping in air and agitating, or by chemical means; the cheapest and best of the latter is found to be permanganate of potash, about one-eighth to half a pound per ton of dry slimes was found to be sufficient. Speaking generally, old slimes are considerably more acid than old tailings, and a consumption of from 8 lbs. to 20 lbs. of lime per ton of dry slimes is by no means uncommon. The use of air is far cheaper, and in some cases it is an absolute necessity, as the gold simply will not dissolve until after aeration, so that, instead of merely accelerating the action of the cyanide, it becomes as necessary as that substance itself. After a few remarks the discussion of the paper was postponed and the meeting adjourned.

NOTICES OF BOOKS.

Patents for Inventions. Abridgments of Specifications
Class 1.—Acids, Alkalies, Oxides, and Salts. Inorganic Period, A.D. 1884—88. Published at the Patent Office 1896. Price 1s.

THE Patent Laws of this country make no provision for an official search as regards novelty, and all patents are taken out at the risk of the inventors. It is therefore incumbent on any person desiring to obtain a valid patent for an invention either to cause a search to be made, or himself to make a search as to the novelty of his invention. By omitting such a search, many a patentee has found, after paying his fees, that his treasured patent is worthless, because it has been anticipated. Of course, in this case the first applicant or patentee possesses all the Patent rights, and the second one has absolutely no rights at all.

A complete and exhaustive search through published Specifications of Patents is a task of considerable difficulty, even for the trained expert with all the resources of the Patent Office Library, for at this moment the number

of printed Specifications of Patents is well over a quarter of a million.

A series of Indexes and Abridgments has been published by the Patent Office as a guide to the specifications themselves, and is freely distributed to the principal public libraries in this country. The Abridgments give a general description of the nature of every invention patented, and the object of their publication is to enable the would-be patentee to carry out, at any rate in some cases, what may be termed a *fireside search*. By the study of these Abridgments he will generally be able to select certain inventions which have already been patented, and which resemble his own invention sufficiently to render it desirable for him to examine their specifications in detail. A printed copy of any specification can be obtained at an inclusive price of 8d., through any Post Office, by a special Postcard (Patents Form C¹). The Abridgments are published in volumes, each volume dealing with one particular class of inventions, such as "Steam Engines," and "Cooking and Kitchen Appliances, &c." for a period of some years. The volumes up to 1877 are not illustrated, and all the subjects have not yet been dealt with, but from 1877 onwards a systematic series, very fully illustrated, is now in course of publication at a uniform price of one shilling per volume (including inland postage). The volumes for the periods from 1877 to 1883 and from 1884 to 1888 have been completed, those for the periods from 1889 to 1892 and from 1893 to 1896 are in active preparation, and later volumes will follow in due course. For the purposes of the Abridgments the whole field of invention has been divided into 146 "Abridgment Classes," and the list of these classes in itself shows what an enormous field this is, and how greatly its products vary. Every triumph of applied science, such as the locomotive, the telegraph, and the dynamo, is to be found here, and every one of our great national manufactures and industries finds its appointed place. Each volume contains abridged descriptions of the inventions falling under one of the 146 classes during the period of which it treats (illustrated by diagrams or drawings wherever possible), a detailed Index to the inventions according to their subject-matter, and an Index to the names of patentees or applicants.

For the use of those who desire to make a careful study of Patents, the Patent Office also publishes an "Abridgment-Class and Index Key" (price 1s., parcel postage 5d.), which shows in detail how inventions are classified, abridged, and indexed throughout its publications.

X Rays in Surgery. ("Razele X in Chirurgie.") By Prof. C. SEVEREANU. With 26 Radiographic Plates. Bucharest: F. Göbl. 1897. Pp. 94.

THIS latest contribution to our knowledge of X rays goes over, to a great extent, the same ground as the earlier books on the same subject.

In discussing the theory of the origin of the X rays, the author is of the opinion that they do not emanate from the cathode itself; that is to say, they are not cathode rays proper, but they are set up by the impinging of the cathode rays on some other body, either the glass envelope of the Crookes tube or the anti-cathode.

The illustrations are very good, and include a number of deformities, fractures, and freaks—such as a hand with six fingers, a foot with six toes, and such like.

The Chemical Composition of Waters. ("La Composicion Química de las Aguas.") By JUAN J. J. KYLE, D.Sc., Professor of Chemistry at the University of Buenos Aires. Buenos Aires: 680 Calle Peru. 1897.

WE find in this pamphlet a large number of analyses of water from all parts of Brazil; some are ordinary potable

waters, but many contain a large quantity of dissolved salts. The results are expressed in parts per 100,000, and, though they represent a great amount of time and work, they are of no particular interest to English readers.

Manuali Hoepli.—*Manual for Chemists and Industrialists.* A Collection of Tables of Physical and Chemical Data, and of Processes of Technical Analysis. ("Manuale del Chimico e dell' Industriale." Raccolta de Tabelle, di Dati, Fisici e Chimici, e di Processi d'Analisi Tecnica ad uso di Chimici Analitici e Tecnici, &c.). By Dr. LUIGI GABBA. Second Edition, Enlarged and Enriched with the Analytical Tables of H. Will. Milan: Ulrico Hoepli. 1898. Pp. 442.

THE physical tables are extremely copious, and are satisfactory in every respect save the prominence awarded to the hydrometric scale of Baumé, which seems to take its stand on its essentially irrational character.

There is a useful table of molecular weights and solubilities in cold water, boiling water, and alcohol.

The fourth chapter, headed "Applied Chemistry," includes the chemical examination of potable waters. Here are given the chief components of the subsoil water of Milan; the regulations for the analysis of potable waters in Paris; directions for the assay of ores and metals, including pyrites, burnt ores, ores of copper, zinc, and lead; the electric assay of metals and alloys. Then follow assays concerning the heavy chemical industries, such as the analysis of the gases of pyrites kilns, and of chamber and tower gases.

According to Wedding 1 Hefner = to approximately $\frac{1}{10}$ Carcel, the luminous power of an Auer gas burner on and after 400 hours is the loss of about 25 per cent.

The assay of soaps, of wax, of mixtures of wax and tallow, and of spurious butters, is fully discussed.

Methods are given for determining the acidity of oils, for detecting cotton-seed oil, colza, sesame, and arachis.

After instructions for the examination of varnishes, we come to the assay of soaps, of textile fibres, tissues, and paper; and the analysis of soils, manures, &c.

The work is exceedingly rich in valuable and rare information. We can especially recommend it to the tropical and subtropical farmer and explorer.

Manuali Hoepli.—*La Grande Industria Chimica.* La Fabbricazione dell' Acido Solforico et dell' Acido Nitrico del Solfato Sodico, dell' Acido Muriatico. By Dott. V. VENDER, Chimico Consultante a Milano, formerly Chemist and Director of Water Works. With 107 Cuts and many Tables. Milan: Ulrico Hoepli. 1897. Pp. 279.

THE volume before us belongs to the useful series of Manuals now being produced by the firm of Hoepli, and treats of the most important branches of heavy chemical industry. The work opens with statistical generalities, which confirm the statement that among the chemical industries sulphur plays a part analogous to that held by iron in the mechanical world. In 1890 the world's production of sulphuric acid amounted to 2800 thousand tons, of which 870,000 were manufactured in Britain, 506,000 in Germany, and 234,000 in France. Italy contributes to the sum-total 120,000 tons. The American yield is not quoted. Among the various qualities of pyrites quoted, the highest grades are those obtained from Spain and Portugal.

The approximate table of atomic weights is, in one case at least, misleading, since it ranks platinum higher than gold.

The only objectionable feature in this work is that it persists in the exclusive use of the hydrometric or areometric scale of Baumé—distinguished for nothing but its disadvantages.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 11, September 13, 1897.

Electrolytic Separation of Nickel and Cobalt from Iron. Application to the Determination of Nickel in Steels.—O. Durn.—Will be inserted in full.

Contributions to the Determination of Iron in Urine.—Dr. Adolf Jolles.—Hamburger's volumetric procedure has been modified so as to require less time. For the gravimetric determination nitroso- β -naphthol is recommended as a suitable reagent. The iron is separated from a hydrochloric solution by a concentrated solution of nitroso- β -naphthol in acetic acid at 50–52 per cent. The precipitate has the composition $C_{10}H_6O, NO_3Fe$.

Simplification of the Determination of Potassium.—Adolf Meyer.—The reagents necessary are normal barium chloride, normal oxalic acid, solution of platinum chloride containing 1 grm. Pt in 10 c.c. free from nitric acid, and lastly alcohol at 80 per cent by volume.

Iodine Number of Cacao Butter.—Dr. D. Held.—The value 51 is admittedly an error.

Determination of Boric Acid according to Gooch.—K. Kraut.—This paper requires the accompanying illustration.

No. 12, September 20.

On Oxycellulose.—L. Vignon.—Under the microscope oxycellulose appears to consist of very short filaments. It turns yellow at 100° and is insoluble in neutral reagents. It is turned blue by iodine and sulphuric acid. The colouration is more rapid and more distinct than with cellulose. The centesimal composition is—

Cellulose. Oxycellulose.			
C	44.44	43.55	
H	6.17	6.03	
O	49.39	50.42	

But if we submit oxycellulose to Lang's procedure (fusion with potassa at 180°) we find—

	Cellulose. Oxycellulose.	
	P.c.	P.c.
Soluble in fused KOH	12	87.58
Insoluble	88	12.42

We are led to consider this oxycellulose as a mixture of 75 per cent oxycellulose and 25 cellulose. The heat of combustion is as follows:—

Cellulose	4224 to 4190
Oxycellulose	4133 „ 4124

Absorption of Basic Colouring Matters.—Cellulose and oxycellulose have been compared from this point of view by steeping in baths of known strength, obtained with safranine and methylene blue, for thirty minutes at ebullition. Their impoverishment serves to measure the absorption. Schiff's reagent (magenta and sulphuric acid), prepared according to Villiers and Fayolle, yields, with oxycellulose, an intense violet colouration. It possesses, therefore, aldehydic functions.

On Retamine.—J. Battandier and Th. Malasse.—Retamine is capable of yielding neutral salts containing two mols. of monobasic acid or one mol. of bibasic acid to one mol. of alkaloid, and basic salts containing one mol. of monobasic acid to one mol. of alkaloid.

Influence of Colouring-matters upon the Fermentation of Highly-coloured Red Wines.—P. Carles and G. Rivière.—The incomplete transformation of sugar in highly-coloured musts is due to the colouring-matter, and

not to the acidity; for decoctions of elder, whether acidified or not, give the same results. This colouring-matter, allied to the tannins, acts as an antiseptic upon the microbia of fermentation.

No. 13, September 27.

Stability of the Phosphorescent Strontium Sulphides.—J. R. Mouret.—In general the phosphorescent strontium monosulphides are of little stability. Like the alkaline or alkaline-earthly sulphides, they have a tendency to become partially polysulphurised or sulphatised, forming hydrosulphates of sulphides. These properties, common to combination of sulphur with various metals, have no direct influence on the phosphorescence of strontium sulphide.

On Parastannyl Chloride.—R. Engel.—The facts summarised in this paper, and which will be developed in a subsequent memoir, readily explain the contradictions of former authors. The so-called *metastannic acid*, obtained at a low temperature, is in reality a mixture of stannic and metastannic acids.

On various Double Chlorides formed by Cinchonamine.—Léon Boutroux and P. Genvresse.—A knowledge of the facts observed by the authors permits us to avoid a cause of error in the detection of nitrates by cinchonamine.

Journal de Pharmacie et Chimie.
Series 6, vol vi., No. 4.

Histological and Chemical Study of the Action of Antiseptics on Muscular Fibres.—A. Riche.—This research is exclusively devoted to the action of antiseptics in the preservation of meat, in connection with a certain very dilute liquid; analysis has shown that it is formed of nearly pure bisulphide of lime. It is slightly acid, and gives off a slight smell of sulphurous acid; similar solutions of the same density were made from commercial sulphites of lime and soda. The matters examined were from the fillet of beef. Experiments were made by immersion in water, and in the reagents for seven hours, and the results compared. After immersion for thirty hours, the fibres were dissociated mechanically and examined under the microscope, and very important differences were found. Measurements of the diameters of the fibres were made, and there was found to be a remarkable difference between those treated in these three solutions and others treated with other reagents, such as boric acid, formol, &c.

Action of Iodine on Albumenoid Matters.—E. Lepinois.—The author considers that iodocasein might be susceptible of a therapeutic application in the case when thyroiodine and other iodised substances have been used or recommended.

On Aloines.—E. Léger.—Aloines have been derived from aloes from many parts of the world, but they may all be divided into two groups: the first comprises, according to various authorities, barbaloin, socaloine, zanaloin, and curacaloine; and the second only nataloin. This latter differs entirely from the others by its almost complete insolubility in water, even when warmed. It is also very slightly soluble in alcohol. According to some writers there appears to be no doubt that the first-mentioned four are identical.

Bulletin des Travaux de la Société de Pharmacie de Bordeaux. August, 1897.

Hygienic Value of Table Mustard.—P. Carles.—Not suitable for abstraction.

New Practical Apparatus for Lixiviation.—L. Barthe.—The apparatus proposed by M. Fonze-Diacon

is incontestably superior to that of Soxhlet, but it is too difficult and expensive to make and too fragile for general use. The author therefore recommends one that is very simple in construction and easy to use. A small tube, contracted at the lower end, is placed inside another of similar shape but larger, but they are prevented from touching closely by a thin glass rod which is placed between them: the large tube is fitted to a flask by means of a cork, and a vertical condenser is connected to its upper end; the vapour of the solvent passes through the interstices between the two tubes, and being condensed, falls down on to the substance to be lixiviated in the inner tube.

Coefficient of Partage of the Monobasic Fatty Acids of the Series $C_n H_{2n} O_2$ from the Condensation of C_1 to the Condensation of C_7 inclusive.—Th. Garraud.—A long paper, not suitable for abstraction.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xvii.-xviii., No. 15.

On the Oxidising Power of Manganous Salts, and on the Chemical Constitution of Laccase.—G. Bertrand.—Continuing his researches on this subject, the author finds that bioxide of manganese, stable in acidulated water, is reduced as soon as hydroquinone is added, giving a manganous salt, at the same time forming quinone. It follows from this that a definite weight of a manganous salt ought to oxidise, at the expense of the air, an unlimited amount of hydroquinone, or any other body equally oxidisable. The principal interest in the observations made in this paper is to complete, so far as concerns the chemical constitution and the mode of action of oxidases, an idea which sprung from the researches the author has already published, on the intervention of manganese in the oxidations provoked by laccase.

Existence of a Proteic Body foreseen by M. Bertrand in the Constitution of Oxidases.—J. de Rey-Pailhade.—The substance discovered by the author in 1889, and called by him "philothion," possesses the properties required by M. Bertrand.

On Cryoscopy of Milk and Organic Liquids.—A. Ponsot.—Two solutions having the same coagulating point are not always equimolecular, and two solutions having the same point of congelation, under conditions of a given pressure, are in osmotic equilibrium under this pressure at the temperature of congelation; but they are not so as a rule at all temperatures, and in particular at the temperature of the organism.

On the Estimation of Caffeine in Coffee.—E. Tassilly.—The methods of estimating caffeine in coffee may be divided into three groups:—1. Exhaust with warm chloroform alone, in the presence of a base, such as lime, magnesia, ammonia. 2. Exhaust with warm water, and, after various treatments, the aqueous solution is agitated with chloroform, or evaporated with magnesia and treated by the same warm solvent in a digester. 3. The coffee alone or mixed with an alkali (lime or magnesia), is exhausted by a solution of an organic salt (benzoate or salicylate of soda), then the mass is exhausted by cold chloroform. These three methods are briefly described and criticised, and the author concludes that the methods of No. 1 group generally give but uncertain results; the methods by using benzoate or salicylate of soda give a very pure product, but it takes up too much time; but none of them are entirely satisfactory.

On a New Method for the Estimation of Caffeine in Coffee.—E. Tassilly.—The author proposes to treat the coffee with water, and then evaporate to dryness; the residue is then treated with sulphuric acid, and the caffeine dissolved in boiling water; at this point one can either evaporate to dryness in the presence of sand and

magnesia, followed by an exhaustion by warm chloroform in a digester, or, add ammonia and exhaust with cold chloroform in a flask.

On the Estimation of Oxide of Iron and Alumina in Phosphates.—N. Blattner and J. Brasseur.—Already inserted,

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1978.

THE PERMEABILITY OF ELEMENTS OF LOW ATOMIC WEIGHT TO THE RÖNTGEN RAYS.

By J. H. GLADSTONE, D.Sc., F.R.S., and
W. HIBBERT, F.I.C.

In the CHEMICAL NEWS (vol. lxxiv., p. 235) there was published a condensed account of a communication made by us to the British Association, on the action of metals and their salts on the Röntgen rays. On December 18th (lxxiv., 298) appeared a longer paper, by Dr. Waddell, on the same subject; and in the CHEMICAL NEWS of October 1st of this year (lxxvi., 161) there appears a second paper which Dr. Waddell sent to Toronto. In this he attacks our general conclusions, specially with reference to the order of permeability of the alkaline metals lithium, sodium, and potassium.

While Dr. Waddell's knowledge of lithium is entirely derived from its salts, we determined its permeability in comparison with sodium and potassium from plates of the metals of known thickness. We thus got rid of any error due to the presence of the acid radicals.

Marangoni has repeated our experiments with larger quantities of the three metals, and come to the same conclusion as ourselves, namely, that lithium has an extremely small absorption. He finds that the metallic lithium must be ten times as thick as the sodium to produce the same absorption.

In judging the statements of Dr. Waddell and ourselves, it must be borne in mind that when we give the order of comparative permeability, we mean the permeability of an equal number of atoms, that is, equal atomic weights,—seven parts of lithium compared with twenty-three of sodium and thirty-nine of potassium, whereas Dr. Waddell compares equal absolute weights.

It is clear that in his method of observation the quantity of the acid radical cannot be the same in the salts which he compares. Yet the effect of this acid radical is very important. He does not seem to have perceived this at first. Even when further experiment gave him results which "surprised" him, he still retained in large part the opinions previously formed.

When the comparison is made for equal thicknesses of the three alkaline metals (not their salts), the order of permeability is still decidedly lithium, sodium, and potassium.

On attentively reading Dr. Waddell's last paper, we do not see that any one of his experiments is inconsistent with our conclusions, but in many cases our conclusions throw light on his difficulties.

The most original part of Dr. Waddell's paper is that on the peculiar granular appearance presented by the shadows of salts in his pill-boxes. We have frequently noticed the same phenomenon, but have never seriously investigated it.

We hope shortly to publish quantitative measurements of the intensity of some of these Röntgen ray shadows, a small number of which were in fact communicated to the British Association at Toronto.

Chemist to the London County Council.—At the recent meeting of the London County Council, the recommendation of the General Purposes Committee, that Dr. Frank Clowes, of Nottingham, be appointed successor to Mr. W. J. Dibdin, F.I.C., F.C.S., as Chemist and Superintending Gas Examiner, was unanimously confirmed.

FURTHER EXPERIMENTS ON THE LIQUEFACTION OF FLUORINE.

By M. MOISSAN and J. DEWAR.

In May, 1897 (*Comptes Rendus*, cxxiv., 1202) we had the honour of presenting to the Academy our first experiments on the liquefaction of fluorine; we will now describe some fresh experiments we have made on this subject.

Liquefaction of Fluorine.—Our latest experiments on liquefaction were carried out by means of an apparatus similar to that which we have already described,—that is to say, a glass bulb fused to a platinum tube, which contained another similar smaller tube; but each of these tubes was fitted with a screw valve, in such a manner that, at any moment, communication—either with the outer air or with the current of fluorine—could be interrupted. This little apparatus was placed in a cylindrical glass receptacle with double sides, containing liquid air. The whole was connected with a vacuum pump, and furnished with a manometer.

In a series of preliminary experiments, we determined exactly the boiling-points of liquid oxygen at various pressures, as shown by the manometer.

In some former experiments we had shown that fluorine does not become liquid at the temperature of boiling oxygen at the ordinary atmospheric pressure.

We now find that, by repeating the same experiment with freshly prepared liquid air, the fluorine becomes liquid as soon as the air begins to boil, at the ordinary pressure.

We have repeated our former experiment, with liquid oxygen as refrigerant, and, on making a vacuum, we find that the liquefaction of fluorine takes place by the evaporation of the oxygen at a diminution of pressure of 32.5 c.m. of mercury.

From these two experiments we are enabled to state that the boiling temperature of fluorine is very close to -187° .

Experiments on Solidification.—When the little glass bulb was three-quarters full of liquid fluorine, we closed both the valves, and then caused the liquid air serving as refrigerant to boil rapidly, at a diminution of pressure of 72.5 c.m. Under these conditions we obtained a temperature of -210° . The fluorine did not show any sign of solidification, but retained its characteristic mobility.

To complete this experiment it becomes necessary to cause the rapid ebullition of the liquid fluorine thus obtained; we hope to achieve this in future experiments. When we had repeated this experiment several times, a slight accident occurred to one of our little instruments containing the fluorine. The screw of one of the valves becoming worn, allowed the air to enter the bulb. This air was immediately liquefied, and in a few moments we had two distinct layers of liquid; the upper, colourless layer, consisted of liquid air; the lower one, of a pale yellow colour, being fluorine.

In another experiment, taking great precautions to prevent the ingress of any air, the fluorine was introduced in its liquid state into a glass tube, the end of which was then sealed before the blowpipe. The sealed tube, containing the liquid fluorine, was kept for a long time at -210° , by the rapid evaporation of a large quantity of liquid air, but it gave no trace of a solid body.

Density of Liquid Fluorine.—To determine the density of liquid fluorine, we brought it into contact with a number of bodies whose density is known exactly. By taking groups of bodies whose densities are very close to each other, it is easy to see which sink and which float in the liquid. This well-known though indirect method was the most suitable for these delicate experiments. We first of all satisfied ourselves that the fluorine had no action on the materials used. To effect this we placed a crystal of sulphocyanide of ammonium (density = 1.31) in a glass

tube surrounded with boiling liquid air; we then turned in, to the bottom of the tube, a current of fluorine gas, by means of a platinum jet. The fluorine was rapidly liquefied, and the sulphocyanide of ammonium was not attacked. We repeated the experiment with a fragment of ebonite ($D = 1.15$), of caoutchouc ($D = 0.99$), of wood ($D = 0.66$), of amber ($D = 1.14$), and of oxalate of methyl ($D = 1.15$). It is of importance, in the experiments we have just mentioned, that the various materials used should be first kept at a temperature of -200° , for some little time.

In one of our experiments a piece of caoutchouc, having been insufficiently cooled, took fire on the surface of the liquid, and burnt completely away with a brilliant flame, without leaving any residue of carbon.*

The experiment was carried out in the following manner:—In a glass tube closed at one end, and of which the lower part had been slightly drawn out, we placed fragments of the five substances we have just mentioned. The tube was then plunged to a third of its length into boiling liquid air. When it was all reduced to a temperature of about -200° we carefully introduced the fluorine gas. This soon became liquefied, and we saw the wood, the caoutchouc, and the ebonite floating easily on the surface of the pale yellow liquid. On the other hand, the oxalate of methyl remained at the bottom, while the amber rose and fell in the liquid, appearing to be of the same density. The apparatus was shaken several times, and the quantity of liquid fluorine increased, but the results were always the same.

We therefore arrive at the conclusion, from these experiments, that the density of liquid fluorine is 1.14. Another point which appears to be of interest is the following:—The fragment of amber floating in the fluorine was very difficult to distinguish, which would seem to indicate that the index of refraction of liquid fluorine is very close to that of solid bodies.

In another experiment we liquefied fluorine in a glass tube which had been previously graduated; we then sealed the tube, which had been weighed before the experiment, and left it alone in a beaker full of liquid air at the ordinary pressure. An hour and a half afterwards, the tube still being in 1 c.m. of liquid air, the fluorine had not changed in appearance. But shortly afterwards, when the air had all evaporated, a violent detonation occurred; the sealed tube and the double beaker in which it had been placed were smashed and reduced to powder. The sealed tube showed us that liquid fluorine sustains, at from -187° to -210° , a diminution of volume of $1/14$ th.

Absorption Spectrum.—We examined with the spectroscopic different samples of liquid fluorine through a thickness of about 1 c.m., either in sealed tubes or by means of our little condensing apparatus, but we have never been able to detect absorption-bands.

Magnetism.—Liquid fluorine placed between the poles of a powerful electro-magnet, does not show any magnetic phenomena. These experiments are the more decisive as we made comparative ones with liquid oxygen, as before; they were repeated several times.

Capillarity.—The capillary constant of fluorine is weaker than that of liquid oxygen. A capillary tube, plunged successively in fluorine, oxygen, alcohol, and water, gave the following figures:—

Height of liquid fluorine	3.5 m.m.
" " oxygen	5.0 "
" alcohol	14.0 "
" water	22.0 "

The Action of some Substances on Liquid Fluorine.

Hydrogen.—Liquid fluorine in a glass tube was cooled down by liquid air boiling at a low pressure. A slow current of hydrogen gas was made to impinge on the sur-

face of the yellow liquid by means of a platinum jet. There was immediate combination, with the production of a flame which lighted up the tube. The experiment was repeated by dipping the platinum jet below the surface of the liquid. At this temperature (-210°) complete combination still took place, with a considerable evolution of light and heat.

In another experiment the hydrogen apparatus terminated with a fine glass tube dipping into the liquid fluorine; when the hydrogen was turned on the combination took place immediately and with violence.

Oil of Turpentine.—Oil of turpentine, frozen and cooled down to -210° , is attacked by liquid fluorine. To perform this experiment we placed a little oil of turpentine at the bottom of a glass tube surrounded with boiling liquid air. As soon as a small quantity of fluorine was liquefied on the surface of the carbide the combination took place with explosive force, a brilliant flash of light, and deposition of carbon. After each explosion, the current of fluorine gas was kept up slowly, a fresh quantity of liquid fluorine was formed, and the detonations succeeded each other at intervals of from six to seven minutes. Finally, after a longer interval of about nine minutes, the quantity of fluorine formed was sufficient to cause, at the moment of the reaction, the complete destruction of the apparatus.*

Oxygen.—The action of liquid oxygen has been studied with much more care, since we observed from our earliest experiments that by passing a current of fluorine through liquid oxygen, we obtained a detonating body.

If we bring a current of fluorine on to the surface of liquid oxygen in a glass tube, the fluorine dissolves in all proportions, imparting a yellowish colour, and giving the liquid a graded tint from the upper to the lower part; the bottom of the tube is hardly coloured. If, on the contrary, we introduce the fluorine gas at the bottom of the liquid oxygen, the yellow colour is produced at the bottom and diffuses slowly to the upper layers.

This phenomenon indicates that the densities of liquid fluorine and oxygen are very near each other. When we have obtained a mixture of liquid oxygen and fluorine, if we allow the temperature to rise slowly, the oxygen evaporates first. The liquid becomes more and more concentrated as to fluorine, and finally the latter begins to boil in its turn. In fact, at the commencement of this boiling the gas coming off will light a match which has only a red-hot point, and will not make lamp-black or silicon red-hot; but, on the other hand, the gas coming off at the end of the experiment will instantly cause these two latter bodies to burst into flame. When the glass bulb is completely empty and its temperature is rising, we suddenly notice a distinct disengagement of heat, and the interior of the glass loses its polish. This rise of temperature is due to the fluorine gas attacking the glass. In this experiment, when using perfectly dry oxygen, no precipitate is produced. If, on the contrary, we use oxygen which has been some hours in contact with the air, the detonating substance we mentioned in our previous communication is produced with great readiness.

In one of our experiments, in which we tried to obtain a notable quantity of this body, we had an explosion strong enough to smash the glass in which the experiment was being performed.

To sum up, this body, which is produced by the action of fluorine on moist oxygen, seems to be hydrate of fluorine, decomposing, with detonation, by a simple rise of temperature.

Water.—We froze and cooled down to -210° a small quantity of water at the bottom of a glass tube. Liquid fluorine formed on the surface of the ice as a mobile liquid without action, and it evaporated on the temperature rising. As soon as the apparatus became warmer the remaining gaseous fluorine attacked the ice with great energy, and we noticed a strong smell of ozone.

* This piece of caoutchouc ran about the surface of the liquid like sodium on water, giving a very intense light.

* In several of our experiments we accidentally let a little liquid fluorine fall on the floor; the wood instantly took fire.

Mercury.—We solidified a globule of mercury at the bottom of a tube. The surface remaining very brilliant, the liquid fluorine surrounded it without causing it to lose its appearance or polish. On allowing the temperature to rise to -187° the fluorine began to boil, the liquid disappeared completely, but the attack of the mercury by the fluorine gas did not take place until the apparatus had almost reached the temperature of the laboratory.

Conclusions.

Fluorine gas is easily liquefied at the temperature of boiling atmospheric air. The boiling-point of liquid fluorine is -187° . It is soluble in all proportions in liquid oxygen and in liquid air. It does not solidify at -210° . Its density is 1.14, its capillarity is less than that of liquid oxygen; it has no absorption spectrum, and it is not magnetic.

Finally, at -210° it has no action on dry oxygen, water, or mercury, but it reacts, with incandescence, on hydrogen and oil of turpentine.—*Comptes Rendus*, cxxv., No. 15, p. 505, 1897.

APPARATUS FOR STUDENTS IN ELEMENTARY PRACTICAL CHEMISTRY.

By GEORGE GEORGE.

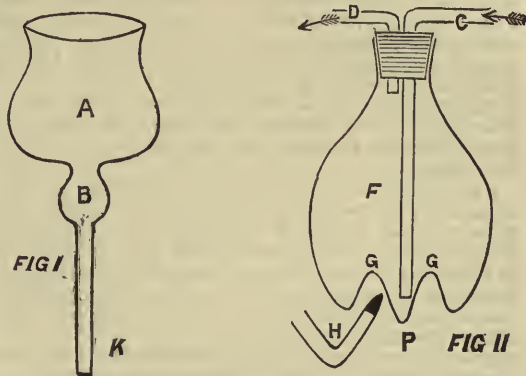
THE teaching of elementary chemistry has undergone a great change since 1889, when the Committee of the British Association made their Report upon the subject.

One of the outcomes of this progressive movement is that the student himself now performs a good many experiments which previously were shown only by the teacher. In consequence, the latter is constantly on the look-out for simple, effectual, and inexpensive apparatus, by means of which a large number of students can simultaneously establish for themselves well-known chemical truths or repeat chemical experiments.

The accompanying diagrams represent apparatus which I have found most satisfactory, and trust other teachers of chemistry may do likewise.

In Fig. 1 is shown a large thistle funnel, used for determining the equivalent weights of the metals, and by means of which boys—using a balance costing about thirty shillings—can obtain results involving less than 0.5 per cent of an error.

The funnel, which has a capacity of about 100 c.c., is made of thin glass, and weighs about 30 grms. B is a small bulb, in which asbestos wool is placed.



The *modus operandi* is as follows:—The bulb B is filled with asbestos wool that has previously been well washed with distilled water and dried. A short glass rod (about 2 inches), rounded at both ends, is then placed with the funnel, and the whole accurately weighed. Then, supposing it is required to find the weight of magnesium which will replace 100 grms. of silver, about 0.15 gm. of magnesium ribbon is weighed out and placed in the funnel A. A short piece of glass rod is fastened to the stem of the funnel at K, by means of a bit of rubber tubing. About 50 c.c. of a solution of silver nitrate (2 per cent) is now poured into A, and the magnesium ribbon well stirred with the glass rod, to remove the silver deposited upon it. After a short time the solution is allowed to run off, by removing the stopper at K. The stopper is then replaced, and a fresh quantity of solution placed in A.

When all the magnesium has been dissolved, the precipitated silver is washed repeatedly with distilled water, and finally with a little methylated spirits. The whole is then removed to the oven, dried, and weighed, the increase in weight representing the weight of the silver deposited.

Of course the same performance can be gone through with other metals and solutions, it being necessary, however, in some cases to heat the solution before placing it in A.

Fig. 2 needs little explanation. It represents a cross section of a flask designed to facilitate the collection of

NOTE ON THE ASSAY OF ELECTRO-PLATING AND GILDING SOLUTIONS.

By ALFRED H. ALLEN.

THE "Note on the Estimation of Silver in Silver-plating Solutions," by Mr. T. J. Baker (*CHEMICAL NEWS*, lxxvi., p. 167), appears to show that very imperfect methods of analysis for the assay of plating solutions are still in vogue. The method employed by Mr. Baker, namely, precipitation of the metals with cyanide by adding nitric acid, and cupellation of the precipitate, will no doubt give accurate results. It appears to me, however, to be less satisfactory on the whole than the following method devised and published by me some twenty years since:—

From 20 to 50 c.c. of the plating solution to be tested is largely diluted with water, and the liquid raised to the boiling-point. Sulphuretted hydrogen is then passed through the liquid, or ammonium sulphide gradually added. The silver falls as a black sulphide, which is liable to be contaminated with copper and zinc. The washed precipitate is rinsed off the filter into a flask or beaker, and treated with an excess of bromine water, which converts it rapidly and completely into argentic bromide. If any sulphur appears to have separated, a drop of bromine should be added to the residue, so as to ensure complete oxidation. Boiling water is now added, and the silver bromide is washed, dried, fused, and weighed. 188 parts by weight of the precipitate represent 108 of metallic silver.

For the determination of the precious metal contained in the solution of the double cyanide of gold and potassium used for *electro-gilding*, I have found the following method very satisfactory:—A measured quantity of the gilding solution is introduced into a porcelain crucible and cautiously concentrated. When in a syrupy condition, a few grms. of pure red-lead or litharge should be added, and the evaporation continued to complete dryness. There is little or no tendency to spitting. The crucible containing the residue is covered and raised for a short time to a moderate red-heat. The lead oxide is reduced by the cyanide present, with production of metallic lead and cyanate, and the reduced metal unites with the gold. The resultant button of metal is separated from the slag, and the gold contained in the alloy isolated either by cupellation or by treatment with pure nitric acid.

Electro-silvering solutions may be assayed in a precisely similar manner; but in this case treatment of the rich lead with nitric acid is, of course, inadmissible, and cupellation must be resorted to.

water produced by a burning jet of hydrogen. The flask F is about 300 c.c. capacity, with rather a wide neck, and at the bottom drawn in all the way round as seen at G G', whilst a cone projects downwards and has its apex at P. Cold tap water is kept circulating in the flask by means of the tubes C and D, so that the cone P is always cold. The burning jet of hydrogen impinges upon this cone, and the water produced is there condensed, drips from P, and is caught in a vessel below. By means of this apparatus, practically the whole of the water produced by the burning hydrogen is collected, and in a short time sufficient can be obtained to determine boiling and melting points, density, &c.

Both of these pieces of apparatus can be made by the teacher himself, if he is at all expert in glass-blowing, or they can be obtained at a small cost from any glass-blower.

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ELECTRICAL ENERGY CAUSED BY THE DIRECT ACTION OF THE ATMOSPHERE.

By H. N. WARREN, Principal, Liverpool Research Laboratory.

THE atmosphere, or, more correctly speaking, the gaseous constituent oxygen contained therein,—not only when in a combined form, as observed in most of the more energetic primary depolarisers, but even in the ordinary gaseous condition,—has, since the introduction of Sir W. Groves's well-known gas battery, been continuously experimented by various scientific men, in the hope of securing at least a somewhat economical supply of that subtle force now so largely employed over the entire field of civilisation.

Experiments of a lengthy nature which have also been engaged in, at the above laboratory, have at last terminated in the successful introduction of an electrical generator, which, even at the present time, although only in its infancy, is more than promising as a liberal source of electricity, not only on account of the extremely low rate of the chemicals employed, but also at the same time ranking next to perpetual motion on account of its perfect resuscitation, whereas the simplicity of its construction enables any one, without any special knowledge, to obtain at a trifling expense an electrical current of almost any required pressure.

The positive element of this powerful generator is composed of plates consisting of a special porous compressed graphite, of which one quarter of the plate is rendered active by immersion in platinic oxalate, and, after drying, igniting the same in an atmosphere of hydrogen gas. By this operation a very finely divided platinum surface is obtained, which, when in contact with a solution of ferrous sulphate, readily induces the oxygen of the atmosphere to combine with and to oxidise the iron present to the state of a ferric salt. In order to construct the cell, several of these so-prepared plates of graphite are attached to a circular lead beam, which surrounds a porous diaphragm containing as a negative element a rod of amalgamated zinc, the carbons being so arranged as to allow the platinised portion to project above the solution consisting of a strongly acidified portion of ferric sulphate. On completion of the circuit a powerful current is at once set free, and continues until the complete reduction of the ferric salt has taken place, which naturally terminates the action. On now withdrawing the zinc from the interior solution, an exactly reverse action is observed, the active platinum surface of the carbon condensing the atmospheric oxygen, which steadily re-oxidises the ferrous salt, and thus renews the action when required. With four ounces of ferric salt, contained in a generator 7 inches by 5, an electric current may be maintained with very slight fall at 2 v. 8 amp. for twenty-four hours.

A further modification of the above cell is now under experiment, in which it is hoped to be able to absorb the hydrogen gas by means of special manganese compounds, thus replacing the more expensive element platinum. For laboratories, and indeed any spot where a long-continued action is required, such as electro-dissolution, they compare very favourably with nitric acid cells, and all other depolarisers which evolve noxious vapours.

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DETERMINATION OF PHOSPHORUS IN STEEL, IRON, AND IRON ORES.

By JULIUS OHLY, Ph.D.

IN the determination of phosphorus according to the molybdate method it has been customary either to obtain the yellow precipitate in the usual manner,—to dissolve it in ammonia water, reduce it by means of zinc, and titrate the ammoniacal solution with permanganate,—or to determine the amount of phosphorus present by means of a standard solution of sodium hydrate. In the first method the only indication for the completeness of the reduction has been found in the colour of the reduced liquid, which is supposed to be of an olive-green colour before the addition of the permanganate is admissible.

It has been found, however, that the operating chemist must be extremely careful and thoroughly experienced, in order to strike this point properly, and even then the results obtained have been found to be wanting. Besides the reducing operation with zinc, &c., is of a very tedious nature, and the tendency has been for these reasons to do away with the reduction of the ammoniacal solution entirely, and to substitute a more speedy and reliable procedure instead.

The following method is considered by the author as an improvement, as it does not leave anything to be desired in speed or reliability, being fully equal in results to those obtained by the gravimetric method.

Two grms. of sample (steel) are dissolved in 45 c.c. nitric acid, sp. gr. 1.16, in an 8-ounce Erlenmeyer flask, by heating until in solution. While hot, 5 c.c. saturated solution of potassium permanganate are added, and the whole allowed to boil until the pink colour has disappeared. Five or six drops of a saturated solution of sugar are then added, and, if these do not dissolve the precipitated oxide, a few drops more are added, avoiding carefully all excess. Let the flask cool now to about 60° C., add 5 c.c. ammonia, shake until clear, add 30 to 40 c.c. molybdate, shake well, allow to settle for a moment, filter with suction, wash with 2 per cent nitric acid six times, and rinse the flask with same. Then wash precipitate and flask with a 2 per cent solution of potassium nitrate, remove filter and precipitate from funnel, and put them back into the flask. Add 25 c.c. standard sodium hydrate solution, shake until the yellow precipitate is in solution, wash sides of flask down with water, add 3 or 4 drops of phenolphthalein, and titrate with standard nitric acid.

The standard nitric acid is made of such strength that 1 c.c. = 0.01 per cent phosphorus, i.e., 100 c.c. nitric acid (1.42) to 11 litres of water.

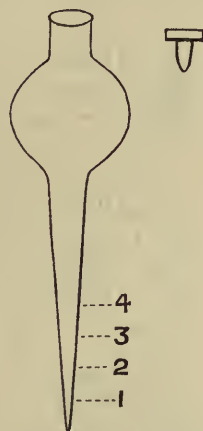
The nitric acid is standardised by running it against a steel whose phosphorus value has been determined gravimetrically.

The sodium hydrate may be of any strength; generally a solution is used of which 25 c.c. = 16 c.c. of the acid. A blank on the sodium hydrate ought to be run every day. The molybdate solution used is made by dissolving one pound of MoO₃ in 1200 c.c. water + 700 c.c. ammonia (0.90). The solution obtained is filtered, and 300 c.c. nitric acid (1.42) added. This forms the stock solution. To 1200 c.c. water and 475 c.c. nitric acid (1.42) add 575

c.c. of the stock molybdate, and use the solution thus obtained for determinations.

The method given above is used for pig-iron and ores with equal success. Unless there is a large percentage of insoluble carbon in the pig-iron it need not be filtered off. For ores it is necessary only that the phosphorus be all in solution in a nitric acid menstruum.

As the most important point to be reached by the chemist in these determinations consists in being enabled to give a reliable result in the possibly shortest time, another process, which gives very satisfactory results for all technical purposes, has also found introduction in some of the most prominent iron and steel works. It consists in the application of Goetz's phosphorus tubes, of the following shape:—



These tubes are charged with the properly prepared solution of iron or steel, as given above, the molybdate solution added, and are then placed into the circular openings of a disk which holds six or more of these filled tubes and rotates on a pivot. When charged it is set in motion by the application of the electric current, the latter giving to the mixture in the several tubes that motion which is generally given by the hand of the operating chemist. The scale, marked at the lower extremity of the tube 1, 2, 3, 4, tells directly the amount of phosphorus indicated by the bulk of the precipitate. The latter settles very rapidly in the lower part of the tube, and becomes very compact, and that to such an extent that the tube may be inverted without the operator incurring any risk of losing a part of the precipitate.

These tubes have been used for all technical purposes with great success for steel and iron exclusively, but there is no reason apparent why they should not be applied for the determination of phosphorus in ore as well.

DISSOCIATION SPECTRA OF SOME MELTED SALTS. METALLOIDS; CHLORINE, BROMINE, IODINE.

By A. DE GRAMONT.

Most melted salts give, under the action of a condenser spark of large capacity (which dissociates them), line-spectra in which each body is represented by the characteristic lines of its own spectrum (*Comptes Rendus*, July 8, 1895). I was by this means enabled to obtain some beautiful spectra of metalloids at the ordinary atmospheric pressure without having recourse to Plücker or Salet tubes.

I will now describe the spectra of chlorine, bromine,

and iodine, obtained by the same method, principally from their alkaline salts, in which the small number of metallic lines to deduct renders their study more easy.

In these experiments I used, for the purpose of holding the fused salts, two thick platinum wires, flattened out at the ends, arranged in V shape, the flattened ends forming the angle, the salts being then kept fused by means of a Bunsen. When the short spark is made to traverse the pasty or melted salt between the wires the air lines disappear. The platinum lines rarely appear, and then only by accident, so long as a trace of the salt remains on the wire. I used a direct-vision spectroscopic with two compound prisms, constructed according to M. Cornu's design. It gives very good dispersion, but is rather absorbent. By removing one of the two systems of prisms, the entire spectrum could be examined, and even then the D line was distinctly doubled. That part of the spectrum between λ 700 and λ 430 was then measured over again with the two systems of prisms. The dispersion thus obtained gave for the lines D_1 and D_2 a separation on the micrometer which could easily be read to the tenth of a degree. The coil gave, without the condenser, and excited by four bichromate cells, a spark 50 m.m. long. The condenser was formed of Leyden jars, each surface of which was about 0.12 square metres, interposed in quantity in the secondary current. In these experiments I found it best to use from four to six of these jars, thus getting a condensing surface of from 0.46 to 0.70 square metres.

Condensers used in previous researches of this nature were much smaller in comparison with the coils used. We can now understand why, in the descriptions of metallic spectra produced from salts (notably Sir J. N. Lockyer, "Studies in Spectrum Analysis," 5th edition, Chap. VI., 1894), we find no mention of the appearance of the spectra of metalloids, which would, however, have been observed if a greater condensation had been used.

The wave-lengths here given were obtained by means of transformation curves of the readings of the scale of the spectroscopic. I made them for one and for two prisms by the aid of the figures given by M. Thalén for the principal lines of the metals, and more especially for those of iron. These numbers were compared with Angström's normal solar spectrum; to compare them with recent observations of the same photographic spectrum by Rowlands, we have only to refer to the table of corrections given in Appendix B of Watts's "Index of Spectra."

It is almost impossible to obtain in the ordinary work of a chemical laboratory a greater degree of accuracy than I have attained in the measurements of wave-lengths, even when giving, as I have, the averages of twenty readings in series of different observations.

If it is in the salts of the alkaline metals that one seeks the spectra of the metalloids, and this seems to be most probable, we should at first sight recognise the principal lines of potassium and sodium by simple comparison with Plate V. of M. Lecoq de Boisbaudran's beautiful Map of Luminous Spectra. We should also be able to study them with advantage in the alkaline carbonates; for, under the conditions of the experiment, these latter only give as carbon lines, and those very faintly, C α 658.3 and 657.8, in the neighbourhood of the red hydrogen line (the first hardly visible, even doubtful), and C β 426.6 in the indigo. It is, further, necessary to bear in mind the fact of the widening of the lines of the alkaline metals—the great condensation of the spark used makes this so diffuse as to almost completely efface the faint lines, leaving hardly any but the following lines visible:—

For sodium—

Na δ (616.0) (615.4), Na β (568.7) (568.2),
Na γ (497.9) double, Na α (589.5) (588.9),
Na ϵ (515.5) (515.2) diffused.

TABLE I.—Chlorine.

Chlorine (from melted chloride). A. de Gramont.		Hasselberg.	Free chlorine (in tubes). Salet.	
—	Not seen.	—	675	} A band divisible into four lines.
—	Doubtful in the salts, T.	—	667	
Cl α	610.5	—	610.7	} Quite visible.
1. 545.6	Well marked, T.	545.67	546.0	
Cl β	2. 544.3	544.36	544.5	} Strong.
3. 542.3	" " T.	543.40	542.3	
4. 539.2	" " T.	539.24	538.9	
532.5	Very faint, and of uncertain origin.	—	—	
—	Not seen.	528.47	—	} Very strong ; double.
Cl γ	522.0	521.98	522.0	
521.6	Strong, T. } First principal group. }	521.62	521.6	
—	Not seen.	518.88	—	
—	" } Seen by Thalén.	517.22	—	
—	" }	516.08	—	
—	" }	511.28	—	
Cl δ	1. 510.3	510.24	510.2	} Double.
509.9	" T.	509.82	509.8	
2. 507.7	Strong, T.	507.76	507.9	} Strong.
499.4	Fairly strong, T.	499.77	499.3	
497.3	Rather faint, T.	497.24	497.2	} Double.
—	Not seen.	494.53	—	
—	Not seen.	493.79	—	} Double.
Cl ϵ	1. 492.4	492.53	?	
491.6	Very plain, T.	491.72	491.8	} Strong.
2. 490.3	Fairly strong, T.	490.44	490.3	
3. 489.7	" T.	489.69	489.6	} Strong.
Cl ζ	1. 482.0	481.98	482	
2. 481.0	" T. } Second principal group }	480.97	481	} Strong.
3. 479.4	" T. }	479.30	479.3	
478.1	Well marked, T.	478.08	478	} Diffused.
476.8	" T.	476.90	477	
474.0	Faint.	473.97	473.5	} Band.
Cl η	457.2	—	457.8	
Cl θ	—	—	436	} Diffused.
—	"	—	434.5	
—	"	—	432	} Diffused.
—	"	—	431	
—	"	—	427	} Band.
—	"	—	425	
Cl ι	—	—	413.3	Band.

TABLE II.—Bromine.

Melted bromides (A. de Gramont).		Free bromine in tubes (Salet).	
—	—	700	approximately.
—	—	678	"
—	—	663	"
—	—	658.2	"
—	—	655.8	"
—	—	654.5	"
Br α	635.15	635.2	Fairly good.
Br β	614.6	614.7	Small group; the widest line, and the least refrangible.
—	587.0 ?	587.5	"
Br γ	582.95	583	Double; the strongest and the most refrangible.
Br δ	571.9	572.2	Double; the most refrangible.
Br ϵ	558.95	559	} Double; the most refrangible; fairly strong.
551.0	Well marked; appears single, T.	550.8	
549.75	" T.	549.6	} Resoluble band.
549.1	" T.	549	
544.7	Weak, often hardly visible, T.	545	} Resoluble band.
543.5	Fairly good, T.	542.2	
542.4	Good; double ?, T.	532.6	} Strong.
1. 533.1	Very strong, T.	530.4	
2. 530.4	Strong, T.	527.3	} Strong.
—	—	526.5	
Br ζ	3. 523.65	524.0	} Strong.
4. 518.25	Very strong, T.	518.3	
5. 516.4	Well marked, T.	516.5	} Strong.
510.65	Fairly strong; variable; of doubtful origin; seen by Plücker.	—	
Br η	505.4	506	Well marked, T.

TABLE II.—*Bromine (continued).*

Melted bromides (A. de Gramont).		Free bromine in tubes (Salet).	
Br θ	492.9 Fairly strong, T.	493	Easily visible.
Br μ {	1. 481.6 Very strong, T.	481.5	"
	2. 478.6 " T.	478.7	Fairly strong.
	476.7 Easily visible, T.	476.6	
	474.3 " T.	474.2	
	472.0 " T.	472.0	
Br ν	470.35 Very strong, T.	470.4	Fairly strong.
	469.15 Fairly visible; often weak.	467.6	
	467.65 Fairly strong, T.	461.7	
	462.2 Easily visible.	454.2	
		436.5	
Br π	436.5 "	428.7	
		423 about	} Vague.
		418 "	
		398	
Br ρ			

TABLE III.—*Iodine.*

Melted iodides (A. de Gramont).		Free iodine in tubes (Salet).	
I α	625.7 Fairly well marked.	625.7	
	620.6 " "	621.0	
	612.6 Very well marked.	612.5	Easily seen.
I β {	608.5 Fairly visible.	597.8	Double.
	607.8 Fairly strong.		
I γ	595.0 Strong.	595.2	Fairly strong.
	578.8 Fairly visible.	579	
	1. 577.45 Well marked.	577.3	Easily seen.
I δ {	2. 576.0 Fairly well marked.	576	
	3. 573.75 Well marked.	573.8	Fairly strong.
	4. 570.9 " "	571.1	"
	569.1 Fairly well marked.	568.8	Easily seen.
	5. 567.75 Well marked.	567.3	Fairly strong.
I ϵ	562.6 " "	562.4	"
	561.3 Very faint.	561	
	560.1 Faint (seen by Plücker).		
	559.5 Faint.	559.6	
	555.3 Very faint.		
	552.4 " "		
	550.7 Faint.	550.1	
	1. 549.8 Strong.	549.4	Fairly strong.
	2. 549.5 Fairly visible.	546.1	Strong.
	3. 546.5 Very strong.	543.3	Easily seen.
I ζ {	4. 543.7 Strong.	540.3	Strong.
	540.4 " "	536.6	
	536.9 Fairly strong.	534.4	Fairly strong.
	1. 534.3 Very strong.	533.6	"
I η {	2. 533.65 Very strong; almost coincides with Cd No. 3.		
	526.7 Easily seen.		
	526.35 Fairly visible.	526.5	
I θ	524.45 Well marked.	524.3	
	521.55 Well marked; almost coincides with Cl γ (521.6).	521.5	
	517.55 Faint.		
I μ	516.15 Very strong.	516.2	Strong.
I ν	510.6 Well marked; variable.		
	506.5 Fairly visible.	506.5	
I ξ {	486.4 Easily seen; diffused.	497.5	
	484.96 " "	486.5	Easily seen.
	480.45 Faint.	485	Faint.
	476.5 " "	480.3	Very faint.
	473.15 " "	476.3	"
	467.65 Well marked; almost coincides with Cd No. 6.	472.9	"
I $\omicron$ {	466.7 Well marked.	467.7	Easily seen.
	464.15 " "	466.8	"
	463.1 " "	464	
I π {	462.2 " "	463.4	
	445.35 Fairly well seen; very diffused.	462	
	444.9 Fairly visible; very diffused; often confounded with the previous one.	445.6	
I ρ {	443.9 Rather faint; diffused.	443	
	441.1 Faint; diffused.	444	
		441	
I σ	422.45 Very well seen.	440	
		442	

And for potassium—

K δ (769.8) (766.5)	K β (536.0) (534.4) (534.0)
K γ (693.9) (691.1)	K ζ (482.8)
(630.8)	(426.4)
(611.7)	(418.5)
K α (583.2) (580.1) (578.3)	K ϵ (404.5)

The alphabetical designations which precede are from M. de Boisboudran's map, and the figures are those which I obtained.

The spectra of free chlorine, bromine, and iodine are those which have up to the present been principally studied first by Plücker and later by Salet. The former made some excellent maps, but did not make any direct measurements of wave-lengths; these have, however, been deduced approximately by Watts, and will be found in his "Index of Spectra."

M. Salet then took up the question, and measured the wave-lengths obtained by a spark traversing a tube containing the free metalloid at ordinary pressure. He has since made some corrections in these figures, and I have reproduced them in this paper to facilitate comparison with those obtained by me.

For free chlorine only, more precise measurements than those of M. Salet have since been made by Thalén and Hasselberg, and I have thought it worth while to give M. Hasselberg's figures as being more recent. The lines marked T were observed also by Thalén. (See Table I.)

The following lines seen by Thalén in free chlorine were not perceived either by Salet, Hasselberg, nor by me in melted chlorides:—

559.35, 552.77, 535.55, 533.20, 531.25, 520.55, 517.40, 514.20, 503.05, 502.05, 477.35, 470.45, 469.80, 466.00, 464.80, 464.00, 463.80, 460.80, 459.60, 459.05, 452.70.

All the chlorides tried gave, with the greatest distinctness, the chlorine lines—with the exception, of course, of those which were effaced by their closeness to, or apparent coincidence with, the intense metallic lines. In a general way we may add that the entire spectrum of chlorine is much finer, the lines are brighter and sharper in the melted chlorides with a condenser spark than with free chlorine in tubes. The most characteristic groups of chlorine in the melted salts are principally Cl γ and Cl ζ , and, secondly, Cl β and Cl δ . The spectrum of chlorine has been examined in this manner in the melted salts of the chlorides of sodium, potassium, lithium, rubidium, cadmium, and zinc.

Bromine.

Since M. Salet's memoir, the emission spectrum of bromine has not, to my knowledge, been the object of any spectroscopic research; its spectrum, much richer in lines than that of chlorine, is as clear and as easily observed in the melted salts. Free bromine gave M. Salet a series of faint lines in the red, from 654 to 700, but I was not able to detect them in the bromides. The most characteristic groups of bromine in the melted salts are principally Br ν , Br μ , Br ζ , ξ ; and, secondly, Br ζ , ξ . The observations have been carried out with bromides of sodium, potassium, cadmium, and zinc. (See Table II.)

Iodine.

Like bromine, the emission spectra of iodine has not been made the subject of any research since that of M. Salet. It is richer in lines than are the spectra of either chlorine or bromine, notably in the orange red. The principal lines in this part of the spectrum, I α , I β , I γ , are easily seen, even with only a small quantity of iodine in the salt under examination. The most characteristic groups or lines of iodine in melted iodides are—Firstly, I η , I μ , I ζ ; and secondly, I ζ (as a whole), I ϵ , I α , I β , I γ . The experiments were performed on iodides of sodium, potassium, and cadmium.

The three spectra of chlorine, iodine, and bromine have their most characteristic lines in the green and the blue, and the complexity of these spectra, as well as their ex-

tension, is increased in the same manner as their atomic weights, and the sensitiveness of the observation appears to be in the same order. (See Table III.).

ON THE COMPOSITION OF CERTAIN CANADIAN VIRGIN SOILS.*

By FRANK T. SHUTT, M.A. F.I.C., F.C.S.,
Chemist, Dominion Experimental Farms.

(Continued from p. 186).

British Columbia.

BEGINNING on the west, or Pacific, coast, your attention is first directed to the tabular statement of the composition of certain typical British Columbian soils. (See Table I.).

These include three well marked groups:—

1. *Deltaic Soils*.—Very rich in plant food. These are formed by the accumulation of detritus, as at the mouths of the Fraser, Pitt, and other rivers.
2. *Valley Soils*.—Largely alluvial as regards origin; rich, as a rule, in both mineral constituent and organic matter.
3. *Bench and Plateau Soils*.—At varying altitudes on the sides and summits of elevations and mountains; variable, but usually light and sandy; of medium fertility, though sometimes very poor.

Possibly there may be other classes of soils in the province, but our investigation has as yet only included those now referred to.

Soil No. 1.—Taken from a valley near Victoria, Island of Vancouver, and representative of a large area that is considered good farming land. When air-dried, it is a dark brown, almost black loam, of excellent texture, homogeneous throughout, and containing clay and humus in good proportions.

In nitrogen and organic matter this soil ranks very high, and—though not as rich in total potash and phosphoric acid as many of our virgin soils—it is by no means deficient in these important constituents.

Soils Nos. 2 and 3.—Represent the soil immediately beneath the preceding sample at the depth of 12 to 18 inches and 18 to 24 inches respectively. In physical appearance and condition, as well as in composition, No. 2 is very similar to sample No. 1; showing that the surface soil has practically a depth of 18 inches. While, as might be expected, the lower sample (No. 3) is somewhat poorer in organic matter and nitrogen, the percentages of potash and phosphoric acid are identical with those in the overlying soil. It is of a yellowish grey colour with streaks of black soil throughout its mass. It will be seen to be of excellent quality for a subsoil.

It will be interesting now to consider the proportions or percentages of these elements that may be looked upon as more or less immediately available for plant use, i.e., the amounts extracted by the 1 per cent citric acid solution before referred to. (See Table II.).

In speaking of minimum limits of available plant food, Dr. Dyer says:—"From a careful consideration of the whole of the results, it would perhaps not be unreasonable to suggest that, when a soil is found to contain as little as about 0.01 per cent of phosphoric acid soluble in a 1 per cent solution of citric acid, it would be justifiable to assume that it stands in immediate need of phosphatic manure."

In potash he obtained results that led him to finally suggest that the limit to be regarded as indicating the non-necessity of the application of special potash fertilisers at 0.005 per cent of potash soluble in the solvent now spoken of.

* Read before the British Association (Section B), Toronto Meeting, 1897.

TABLE I.—Analyses of Soils (Water-free), British Columbia.

No.	Locality.	Surface or subsoil.	Character of soil.	Potash.	Phosphoric acid.	Nitrogen.	Lime.	Loss on ignition (organic and volatile matter).
1.	Victoria, Vancouver Island..	Surface..	Valley soil, black loam	0.23	0.19	0.594	1.29	15.69
2.	Victoria, Vancouver Island..	Depth, 12—18 ins.		0.23	0.19	0.506	1.12	13.61
3.	Victoria, Vancouver Island..	„ 18—24 ins.		0.26	0.12	0.146	1.01	4.63
4.	Alberni, Vancouver Island..	Surface..	Dark red clay loam	0.32	0.08	0.127	1.14	10.79
5.	Alberni, Vancouver Island..	„ ..	Dark red sandy loam..	0.17	0.34	0.163	1.00	11.32
6.	Cowichan, Vancouver Island..	„ ..	Dark red sandy loam, Bench soil	0.39	0.32	0.102	1.37	7.10
7.	Ladners, New Westminster ..	„ ..	Alluvial grey - black loam	0.52	0.28	0.610	0.50	17.25
8.	Squamish, New Westminster ..	„ ..	Valley soil	0.38	0.20	0.091	1.68	3.38
9.	Pitt Meadows, New Westminster ..	„ ..	Alluvial black loam	0.36	0.52	1.050	0.32	31.14
10.	Pitt Meadows, New Westminster ..	Subsoil..	Greyish yellow sandy loam..	0.45	0.13	0.095	0.33	6.37
11.	Agassiz, New Westminster ..	Surface..	First Bench	0.32	0.24	0.159	0.86	6.87
12.	Agassiz, New Westminster ..	„ ..	Second Bench	0.35	0.14	0.101	0.78	4.34
13.	Agassiz, New Westminster ..	„ ..	Valley	0.39	0.18	0.154	0.96	6.92
14.	Agassiz, New Westminster ..	„ ..	Valley	0.35	0.26	0.155	0.97	7.12
15.	Chilliwack, New Westminster ..	„ ..	Valley soil, alluvial	0.63	0.21	0.166	0.98	7.72
16.	Chilliwack, New Westminster ..	Subsoil..		0.51	0.23	0.108	0.90	5.90
17.	Mission, Yale ..	Surface..	Light grey clay loam..	0.45	0.28	0.124	1.86	3.96
18.	„ „ ..	Subsoil..		0.62	0.33	0.076	1.90	3.35
19.	Guisachan, Yale ..	Surface..	Light grey sandy loam	0.32	0.30	0.077	1.22	2.66
20.	„ „ ..	„ ..	Dark „ „	0.53	0.30	0.236	1.70	6.18
21.	„ „ ..	„ ..	„ „ „	0.65	0.38	0.255	1.76	6.59
22.	„ „ ..	„ ..	„ „ „	0.55	0.34	0.259	1.25	7.13
23.	„ „ ..	„ ..	Light grey sandy loam	0.45	0.27	0.045	1.61	2.02
24.	Quesnelle, Cariboo ..	„ ..	Dark grey sandy loam	0.39	0.22	0.399	17.77	12.01
25.	„ „ ..	Subsoil..		0.53	0.19	0.108	3.80	4.60
26.	Cottonwood River..	Surface..	Yellowish sandy loam	0.32	0.34	0.234	1.14	8.28
27.	„ „ ..	Subsoil..	Very sandy..	0.16	0.29	0.057	0.99	3.03
28.	„ House ..	Surface..	Dark grey sandy loam	0.57	0.24	0.412	1.07	13.04
29.	„ „ ..	Subsoil..	Yellowish grey	0.47	0.10	0.050	1.22	3.02

TABLE II.—Comparison of "Available" with "Total" Amounts of Potash and Phosphoric Acid.

No.	Soil.	Potash.			Phosphoric acid.		
		Total potash.	Available potash.	Percentage of total potash available for plant use.	Total phosphoric acid.	Available phosphoric acid.	Percentage of total phosphoric available for plant use.
1.	Surface ..	0.23	0.00483	2.20	0.19	0.01020	5.66
2.	Between 12 and 18 inches	0.23	0.00299	1.36	0.19	0.01055	5.85
3.	Between 18 and 24 inches	0.25	0.00169	0.64	0.12	0.00588	4.90

In available mineral plant food the surface soil now under consideration is seen to give results approximating these limits. The estimations above tabulated are, however, more particularly useful in showing that the upper or surface portions of the soil contain much larger amounts of available food than the underlying soil. We are thus furnished with data to support the view that the greater productiveness of a surface soil, compared with its sub-soil, apart from the presence of nitrogen, in large part is due to the availability rather than to the total amounts of mineral fertilising constituents present.

Soil No. 4.—From Alberni, Island of Vancouver; a clay loam of a deep red colour, masking entirely the presence

of the large amount of organic matter present. This sample is said to represent the soil to a depth of 9 inches over an approximate area of 10,000 acres. The sub-soil of this area is variable, sometimes clay, sometimes gravel and sand. In potash this soil (No. 4) is comparatively rich; in phosphoric acid, however, it is much below the average. As regards nitrogen it is of medium quality.

Soil No. 5.—Also from the district of Alberni, but differing from No. 4 in certain important features. It is known locally as "Fern and Sallal" soil, for the reason that on this virgin soil these plants grow most luxuriantly, crowding out to a great extent other vegetation. At first this soil gives but poor returns, but after several

ploughings—i. e., several seasons working—the yield increases, and good crops are obtained. An examination of the soil showed it to be distinctly acid to litmus paper. There is in this, no doubt, an indication of the cause of the infertility. The effect of exposure to the air through culture would be to correct this sourness, while at the same time locked-up plant food would be set free. Lime and wood ashes have given excellent returns on this soil.

The very large percentage of oxide of iron in these soils—exceeding, frequently, 20 per cent—is a feature worthy of note. It is probable that in the virgin soil a part of this iron is in the ferrous condition, due to the presence of organic matter and to certain other factors. The oxidising of this iron through cultural methods would free the soil of compounds injurious to the tender rootlets of agricultural crops. It is further important to point out that this soil, though yielding 100 per cent of lime to hydrochloric acid, sp. gr. 1.115, had a distinctly acid reaction, and was much benefitted by an application of lime.

Soil No. 6.—A Bench soil, deep red, of sandy character, from Cowichan, Island of Vancouver, similar in appearance to Nos. 4 and 5. It, however, contains less organic matter and nitrogen than these, but is not to be regarded as deficient in any of the essential elements.

A determination of the amounts of available potash and phosphoric acid, ascertained by the citric acid method, afforded the following data:—

Available potash		0.0089
Available phosphoric acid		0.0171

While these amounts do not fall below the limits named by Dr. Dyer, they are, however, such as to suggest that both potash and phosphoric acid would prove beneficial, and give good returns in increased crop yield.

Soil No. 7.—A greyish black soil of excellent texture, from the valley of the Fraser river near one of its mouths, and resulting from the deposition of silt brought down by this river. An area of over 30 square miles is, it is stated, covered by soil of this origin and character. Both from chemical and physical data, this soil would be judged an extremely fertile one, and practical results confirm this opinion. Of phosphoric acid, potash, and nitrogen it possesses quantities considerably above the averages already discussed for fertile soils.

Soil No. 8.—From the Squamish Valley, in the district of New Westminster. The valley is said to have an area of 14,000 acres of arable land. Its sub-soil is clay, though sometimes running into sand. Though containing adequate amounts of mineral food for crop requirements, it is below the average in nitrogen and humus. The ploughing under of green crops—preferably one of the legumes—has been found to improve this soil, both as regards tilth and productive power.

Soil No. 9.—From the Pitt Meadows, New Westminster. An alluvial deposit, composed of the detritus brought down by the Pitt River. It is black loam, in a moderately fine granular condition, and possessing a large amount of vegetable organic matter. On moistening it does not become plastic or sticky, and easily crumbles when dry. The soil granules display a remarkable homogeneity, proving the very intimate incorporation of the vegetable organic matter with the inorganic basis of the soil.

Its mechanical texture seems to be such as would allow freedom for root development, for permeation of air and percolation of water, while at the same time it is sufficiently compact and heavy to prevent easy leaching and to be retentive of moisture.

In potash and phosphoric acid it is seen to be well supplied, comparing most favourably in this respect with soils of great productiveness.

In nitrogen this soil is particularly rich, possessing about 34,000 lbs. per acre, estimating the weight of an acre of soil to the depth of 1 foot to be 3,500,000 lbs. The physical condition of this soil being such that nitrifi-

cation would proceed satisfactorily, the value of this large amount of organic nitrogen becomes obvious.

Soil No. 10 is the sub-soil of the above, and is a greyish yellow sandy loam. From its texture I should expect it to offer a very fair drainage to the surface soil.

Soils Nos. 11, 12, 13, and 14 are surface soils from the Experimental Farm at Agassiz. They are all of medium quality; in tilth rather light, and, though possessing a fair amount of clay, sand predominates. Though not presenting any marked differences, that of the first bench approaches closely in composition to that of the valley soil No. 14. The valley soils are, as a rule, distinctly richer than those occurring at higher elevations.

Soils Nos. 15 and 16 are from Chilliwack, on the Fraser River. They are valley soils, alluvial in origin. While not so rich as the delta soils of the Fraser and Pitt Rivers already discussed, they are by no means poor, possessing a good supply of potash and fair amounts of phosphoric acid and potash. They probably represent more or less truly the character of those soils of medium fertility found in British Columbia in many of her river valleys.

Soils Nos. 17 and 18.—A surface and sub-soil from Mission on Okanagan Lake, Yale district. Both are excellent as regards potash and phosphoric acid, but of poor tilth, caking on being dried into hard masses. The surface soil is somewhat deficient in organic matter, and might be much improved by drainage, judicious culture, and the turning under of a green crop—technically known as green manuring.

Soils Nos. 19, 20, 21, 22, and 23 are surface soils from the ranch of His Excellency the Governor-General at Guisachan. They are sandy loams of varying shades of grey, and, with the exception of Nos. 19 and 23, might be termed, as far as composition is concerned, soils of more than average fertility. These latter are, however, somewhat deficient in humus and nitrogen.

Soils Nos. 24 to 29 inclusive, are from plateaux and upper benches of the Fraser in the Cariboo district, a practically as yet unsettled area. Clover and indigenous grasses of good quality, it is stated, grow well upon them, and the probabilities are that the area here represented will be found suited for grazing purposes. Surface soils Nos. 24 and 28 are particularly rich, judging from the chemical analysis, and should prove very fertile if climatic conditions are favourable.

(To be continued).

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING SEPTEMBER 30TH, 1897.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolis Water Act, 1871.

London, October 14th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 182 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from September 1st to September 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several

samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 182 samples examined during the month all were found to be clear, bright, and well filtered.

The rainfall at Oxford during September was 2.25 inches, the average for 30 years is 2.75 inches, making a deficiency of 0.5 inch. Rain fell on nine days only, but 1.13 inches fell on the 29th. The total excess for this year is now 1.12 inches.

In order to prevent any misapprehension in the public mind with regard to our monthly reports on the quality of the London waters, it may be advisable to repeat that the Water Companies in no way interfere with our position as absolutely independent scientific authorities. Further, they have no information antecedent to publication as to what will appear in our report. Our communications with the Companies are chiefly confined to calling their immediate attention to the least anomaly appearing in the character or the quality of the filtered water; our chief aim being to advise the engineers at the works as to the efficiency of storage and filtration.

There is no city in the world where such minute and incessant care is taken daily and almost hourly to detect and report on the slightest deviation from purity in its water supply.

The following table shows that the water supply of last month was actually in a higher state of purity than it was in the corresponding month of last year.

Comparison of the Averages of the Five Thames derived Supplies for the Months of September, 1896 and 1897.

	Common Nitric Salt.		Oxygen. Per Gall.		Organic Hardness. Degrees.		Organic Carbon. Per Gall.		Carbon. Per Gall.		Colour. Br'n: Blue. Means.	
	Per Gall.	Means.	Per Gall.	Means.	Per Gall.	Means.	Per Gall.	Means.	Per Gall.	Means.	Per Gall.	Means.
Sept, 1896.	2.228	0.834	13.41	0.048	0.087	0.146	14.0	20				
1897.	2.137	0.836	14.25	0.034	0.087	0.104	11.1	20				

Our bacteriological examination of 255 samples taken by us have given the following results; we have also examined 9 other samples, from special points, making a total of 264 in all:—

	Microbes per c.c.
Thames water, unfiltered (mean of 26 samples)	98,485
Thames water, from the clear water wells of five Thames-derived supplies (mean of 126 samples)	72
Ditto ditto	highest 992
Ditto ditto	lowest 2
New River, unfiltered (mean of 26 samples)	387
New River, filtered (mean of 26 samples)	26
River Lea, unfiltered (mean of 26 samples)	3168
River Lea, from the clear water well of the East London Water Company (mean of 26 samples)	36

Apart from the daily bacteriological examination of the clear water wells of the Companies, we frequently make specific tests for the presence of pathogenic organisms. If any other than a negative result had been obtained the fact would have been recorded.

It will be observed in the above table that there was an occasion in which the microbes rose so far above the average. This was quite exceptional, and was connected with the atmospheric conditions following an unusually violent thunderstorm, when 1.13 inch of rain fell in less than an hour. We are in a position to say these microbes were harmless.

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 14, October 4, 1897.

On Antique Glass Mirrors Lined with Metal.—M. Berthelot.—Such metallic mirrors are mentioned by Pliny as manufactured at Brinduvium with an alloy of tin, in which I find the origin of the word *bronze*, which has been so long uncertain.

New Method for the Assay of Metals.—A. Frémont.—The points to be determined are merely the tenacity, the ductility, the fragility, and the homogeneity of the metal in question, and the procedure employed is of course purely mechanical.

On the Photographic "Veil" in Radiography.—V. Chabaud.—The author concludes that—(1). Setting out from a given resistance, the two electrodes of the tube emit alternately cathodic rays, and consequently create two foci. (2). Setting out from the same resistance, the tube emits X rays in all directions; in fact, those rays which take their rise on the second focus do not encounter any obstacle in the tube, and propagate themselves in all directions. (3). A hard tube will require a shorter exposure than a soft tube, but will yield proofs more veiled and less distinct than those furnished by the latter. (4). A voluminous tube with large electrodes will give on the screen a greater luminosity than a tube of small dimensions and small electrodes, but the former gives a less definite image.

Solubility of Liquids.—A. Aignou and E. Duzes.—Not suited for useful abstraction.

Journal de Pharmacie et Chimie.
Series 6, vol vi., No. 5.

Action of Sulphuric Acid on Levo-turpentine.—G. Bouchardat and J. Lafont.—The authors incorporated with essence of French levo-turpentine a tenth part of its weight of sulphuric acid; the product was heated to 150° with excess of potash-alcohol; this was treated with plenty of water. The supernatant oils consist of unchanged turpentine, a little camphene, terpenes, and their liquid polymers boiling at 310° to 320°, and of others solid at a low temperature. The water retains in solution the potassic salts formed by this reaction. These salts were first re-crystallised in water, to eliminate the large excess of alkali, and then in concentrated alcohol. They act on polarised light; they consist of two distinct bodies of the same chemical composition, and have, after repeated crystallisations, been separated. The least soluble of these salts, which we call levo-terebenthenosulphate of potash, is in the form of lamellar crystals, similar to boric acid; its rotatory power in 50 per cent alcohol is $[\alpha]_D = -25^\circ$. The second salt is found in the form of long, felted, silky, anhydrous needles, but its rotatory power is different; under the same conditions of dilution we get $[\alpha]_D = +10^\circ$. The mother-liquors of the preceding salts contain two other saline compounds of a different nature; we are now investigating them.

Composition of Haricots, Lentils, and Peas.—M. Balland.

No. 6.

Estimation of Lime, Alumina, and Iron in Mineral Phosphates.—L. Lindet.—Will be inserted in full.

On the Question of Matches, Phosphorism.—A. Riche.—A long, interesting, historical paper, to be continued, but unsuitable for abstraction.

MEETINGS FOR THE WEEK.

FRIDAY, 29th.—Physical, 5. Professor Stroud will exhibit and describe the Barr and Stroud Naval Range Finder. "A Telemetric Focometer and Spherometer," by Prof. Stroud. Mr. Ackermann will exhibit a Surface Tension Experiment.

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THE CHEMICAL NEWS.

Vol. LXXVI., No. 1979.

THE DISTRIBUTION OF CARBONIC ACID IN THE AIR.*

By W. CARLETON WILLIAMS.

It is well known that the method of estimating carbonic acid in the air, as originally proposed by Pettenkofer, contains a source of error arising from the action of oxalic acid on barium carbonate. Another source of error, due to the action of the baryta on the walls of the glass cylinder, in which the experiment is conducted, has been pointed out by W. Spring (*Mem. Acad. Royale de Belgique*, vol. xxxvii.), and more recently by Letts and Blake (*Chem. Soc. Proc.*, 1896, p. 192).

A series of determinations made by a modification of Pettenkofer's method, in which these inaccuracies were avoided, gave the following results for air collected in the suburbs of Sheffield, one and a half miles W.S.W. of the centre of the town.

	Number of experiments.	Average.	Minimum.	Maximum.
Suburbs	142	3'26	2'16	5'14
Fog	7	3'94	—	—
No fog	135	3'24	—	—
Snow	32	3'58	—	—
No snow	110	3'24	—	—
Rain	—	3'12	—	—
Fine	—	3'14	—	—
Centre of the town	21	3'85	2'80	6'22

The carbonic acid is distinctly higher in the town than in the suburbs. A marked increase is produced by fog

* Abstract of paper communicated to the Commemoration Volume of the University College, Sheffield.

and snow; rain does not exert any decided influence. The observations were made during the months of December to April, and, as far as they go, the results show a maximum of CO₂ in January (3'65), with a steady fall to April (2'64). The carbonic acid diminishes as the temperature rises. This is probably accounted for by the decrease in the consumption of fuel for household purposes during the warmer weather. An increase in the amount of carbonic acid is observed with a very high or very low pressure of the atmosphere.

A difference of opinion appears to exist with reference to the question of the uniform distribution of the carbonic acid in the air of inhabited buildings. In the well-known "Handbook of Hygiene" by Parkes it is stated, on the authority of Lassaigue, Pettenkofer, and Roscoe, that the carbonic acid of respiration is equally diffused through the air of a room. Angus Smith, on the other hand, asserts ("Air and Rain," p. 68) that "the air at the ceiling is generally the worst. This, however, depends upon circumstances; if it has time to cool from the height and space being great, the carbonic acid may be arrested before reaching a great height."

Every-day experience would lead us to suppose that Angus Smith is right: our sense of smell indicates that the air near the ceiling of a room is, as a rule, appreciably less pure than the air four or five feet above the floor.

In order to decide between these conflicting statements, samples of air collected in public rooms, class-rooms in Fifth College, and dwelling-rooms in private houses, were examined. The results clearly show that Angus Smith is correct. In lofty rooms, from 26 to 31 feet in height, the air near the ceiling contained less carbonic acid than the air near the floor, but in rooms of a height between 9½ and 16 feet, the air collected 2 feet below the ceiling invariably contained more carbonic acid than the air taken at 2 feet above the floor. As is naturally to be expected, the difference is greater at night when gas or lamps are burning than it is in the day time. During the day, the mean difference is 1'7 volumes in 10,000, but at night the air at the top of the room contained, on an average, 3'7 volumes of CO₂ in 10,000 in excess of that present in the air 2 feet above the floor. These results show that the distribution of CO₂ in the air of an inhabited room is not uniform. (See accompanying Table).

	Height. Ft.	Ins.	Space. In cubic feet.		° C.	Vols. CO ₂ in 10,000.	
Small class-room	16	0	2656	Ceiling, 11.30 a.m.	17	5'19	} 7 persons. Gas-stove burning. 4 persons. Gas-stove burning.
" " " " " "	"	"	"	Floor, "	16	5'15	
" " " " " "	"	"	"	Ceiling, 3 p.m.	17	10'48	
" " " " " "	"	"	"	Floor, "	16	10'23	
PRIVATE HOUSE.							
Bedroom	11	6	3812	Ceiling, 8 a.m.	14'5	4'15	} 2 persons.
" " " " " "	"	"	"	Floor, "	14	3'51	
Dwelling-room A	"	"	"	Ceiling, 9 p.m.	15	11'28	} Open fire; 3 persons. Open fire; 3 people. 2 gas jets; 1 oil lamp. 3 persons in the room. Open fire, lamp, and candles. Open fire; 3 people. Open fire; 1 person. Open fire; 1 lamp. 1 gas jet; 1 person present. Open fire; 2 people. Open fire; 3 people present. 2 gas jets burning.
" " " " " "	"	"	"	Floor, "	13	9'91	
" " " " " "	11	0	4285	Ceiling, 3 p.m.	17'5	7'09	
" " " " " "	"	"	"	Floor, "	17'25	5'27	
" " " " " "	"	"	"	Ceiling, 9 p.m.	19	16'66	
" " " " " "	"	"	"	Floor, "	16	10'31	
" " " " " "	"	"	"	Ceiling, 8 p.m.	18'2	13'08	
" " " " " "	"	"	"	Floor, "	13'5	11'28	
" " " " " "	9	6	2907	Ceiling, "	20'5	10'45	
" " " " " "	"	"	"	Floor, "	18	7'63	
" " " " " "	"	"	"	Ceiling, 4 p.m.	15	5'59	
" " " " " "	"	"	"	Floor, "	14	5'04	
" " " " " "	10	6	2520	Ceiling, "	16'4	5'31	
" " " " " "	"	"	"	Floor, "	13'6	3'31	
" " " " " "	"	"	"	Ceiling, 8 p.m.	18'4	10'47	
" " " " " "	"	"	"	Floor, "	14'6	5'07	
" " " " " "	9	6	2090	Ceiling, 6 p.m.	14'4	6'62	
" " " " " "	"	"	"	Floor, "	12'5	4'18	
" " " " " "	"	"	"	Ceiling, 7.40 p.m.	18	10'48	
" " " " " "	"	"	"	Floor, "	13'8	5'73	

SEPARATIONS WITH ALKALINE ACETATES.

By HARRY BREARLEY.

(Continued from p. 177).

V. Aluminium and Copper from Iron.

A SOLUTION of aluminium chloride containing considerable free acetic acid cannot be precipitated by an excess of alkaline acetate; and precipitates formed locally are dissolved on immediate stirring, although if such a precipitate is allowed to roll about unbroken it becomes difficultly soluble.

Qualitative tests made some months ago induced one to believe that, as in the case of chromic salts, a very considerable separation of aluminium from iron might be made in presence of large amounts of free acetic acid.

A very short experience, under well-defined conditions showed an altogether unexpected difficulty. An example will readily illustrate the point. Say that a solution containing 1 grm. iron, 0.1 grm. Al, its maximum amount of dissolved hydrate, 10 c.c. acetic acid, and 10 to 12 c.c. acetate, is heated to boiling. A similar solution, lacking only the aluminium, would have its iron precipitated during the operation; but the sample would not show the least trace of turbidity. An additional 10 c.c. of acetate would form a local precipitate, which would be readily dissolved as it moved through the liquid. With about 40 c.c. of acetate the solution would become turbid, but if the boiling were continued for an hour or so there would be no considerable separation of iron. Indeed it would need about 60 c.c. of acetate to separate the iron so far as to produce a colourless filtrate. If the deep-coloured supernatant solution of a partial precipitation is poured on to a large asbestos disc, the filtration is sharply arrested in a manner very suggestive of alumina.

This peculiarity, in a similar degree, is noticeable whether the acetic acid be increased or decreased, and whether the dissolved hydrate be as stated or none whatever. These observations show that the practice, in co-precipitating iron and aluminium, of adding considerably more than enough acetate to precipitate the iron, is a necessary one; they also take the edge off one's surprise at finding only a small percentage of aluminium separated by any modification of the acetate separation.

Any useful separation of Al and Fe by means of alkaline acetates appears to be hopeless. On this account the alumina separated from the filtrate was never quantitatively estimated.

It is only necessary now to remark that, in the presence of large quantities of aluminium, some of the separations previously noticed may not be performed with perfect accuracy. To precipitate 1 grm. of iron, after neutralising and adding 10 c.c. acetic acid, would require—

In presence of 0.1 grm. Al 60 c.c. acetate.

"	0.05	"	35	"
"	0.01	"	20	"
"	0.0025	"	14	"

Luckily the proportion of Al in steel usually falls near the last-named amount, and would therefore occasion no appreciable change. For the sake of completeness, however, it is desirable to enquire how the 60 c.c. of acetate would act in a nickel estimation (say) if as much as 10 per cent aluminium chanced to be present. Two tests, with 0.1 grm. of Ni present, yielded percentage recoveries of 98.0 and 98.4 respectively. A reference to Table XIII. (p. 166) will show that this is a much better result than is obtainable with pure mixtures of iron and nickel. If it be not too great a liberty to say so, it seems as though the additional acetate was too busily engaged with the aluminium to give much attention to the nickel.

A more complete separation of iron and nickel, under like circumstances, might be made by adding large amounts of ammonium chloride. With this modification less acetate would be needed, and, on the authority of

Jewett (CHEMICAL NEWS, xl., 273) a more perfect separation is ensured.

Assuming a perfect separation of Fe and Ni, a substitution of alkaline chromate for acetate would readily precipitate the iron, but a very considerable—how much has yet to be determined—amount of aluminium is to be found in the filtrate. When precipitated as alumina in the subsequent titration, this element would be highly objectionable, not only on account of its interference with the indicator, but also on account of its possible influence on the titration itself.

According to Moore (CHEMICAL NEWS, lxxii., 92), "in presence of alumina, either citric acid, tartaric acid, or soda pyrophosphate, may be employed to keep it in solution." The points raised in these two paragraphs will be considered in a later paper.

Copper from Iron.

The acetate separation of copper cannot be said to have ever been greatly favoured, partly, it may be surmised, because a casual separation would almost certainly be a bad one. Wherever the separation is now used, it is generally accompanied by the volumetric estimation of the copper, without a preliminary separation of the precipitated iron.

The possibilities of acetate separation will be best understood by comparing its behaviour when an excess of acetate is used with one of the previously separated elements under like conditions.

Table XV. sets forth this comparison. The corresponding precipitations were made under as identical conditions as could be maintained in two sets of experiments made so widely apart. Attention is again called to the turbidity temperatures as evidence of the similarity. What difference there is favours the copper.

TABLE XV.

Acetate. C.c.	Percentage recovery of		Respective Temp. turbidities.
	Nickel.	Copper.	
10	100.0	98.0	— 91° C.
20	99.0	92.9	72°, 74° C.
50	95.2	69.6	60°, 62° C.
100	90.0	53.1	53°, 54° C.

Like most of the preceding elements, the separation is increasingly accurate as the free acid rises, and decreasingly so as the acetate rises. Any attempt to increase the volume of acetic acid necessarily entails an increase of the acetate required to effect a separation. So far as copper is concerned this is a disadvantage in that a less perfect separation is obtainable with a minimum acetate, and somewhat of an advantage in that a given excess of acetate causes a slighter decrease in the recovery. This advantage or disadvantage is probably true of nickel, cobalt, manganese, and every other element—except chromium as chromic acid—whose acetate separation from iron is practicable, although none of the preceding metals have been nearly so sensitive as is copper in this respect.

TABLE XVI.—With 30 c.c. Acetic Acid.

Acetate.	Per cent recovery.	Turb. temps.
30 C.C.	95.2	93° C.
50 "	91.2	84° C.

It might be presumed, from the foregoing, that a smaller volume of acid (acetic) with only so much acetate as would precipitate the iron after prolonged boiling would give improved results.

Two samples were boiled—

Minutes.	Per cent recovery.
18	98.6
40	98.8

The filtrate of the first sample was distinctly tinted with

unprecipitated iron. Evidently mere prolonged boiling has no effect on the separation.

To go a step further, and passing over decreasing volumes of acid and acetate, which would give increasingly better results, it might be predicted that Schwarzenberg's or Herschell's method, which consists of boiling a solution containing the maximum amount of dissolved hydrate, would give the best results of all.

Truly it might be objected that this would not be an acetate separation, nor would it, although in a mathematical sense it may be regarded as the limiting case of such separations. In a practical sense, too, it may often be convenient to convert a Schwarzenberg into an acetate separation, when the neutralisation has not been performed with the necessary exactness. The Schwarzenberg reaction is generally assured by adding large amounts of alkaline chlorides. In the two cases given below no such addition was made. In neither case was the solution filterable when the boiling point was reached. I. was boiled five minutes; II. thirty minutes.

TABLE XVII.

	Temp. turb.	Per cent recovery.
I.	81° C.	99.45
II.	83° C.	99.10

These results are particularly noteworthy, because copper is not one of the metals where separation from iron by these means is generally recommended.

The preceding tests were all made with soda salts, so as to accommodate the filtrate to the modified cyanide estimation of copper, an account of which is to be found on page 189.

The presence of soda chloride in unusual quantities rather improves the separation.

Separations made with ammonia salts yield somewhat better results, under like conditions, than do soda salts. The presence of large amounts of ammonia chloride would presumably still further increase the accuracy of the separation.

In the following table there are summarised some results with varying proportions of copper. The separations are made with soda acetate, without any unusual amount of soda chloride being present. For reasons just stated this circumstance places the acetate separation in its most unfavourable light, and is only adopted on account of the greater constancy of the soda cyanide estimation of copper. Many other means of estimating the separated copper are, of course, indifferent to this demerit of the ammonia salts. In case such are allied to the acetate separation, it is well to remember that ammonia salts are better than soda, and Schwarzenberg's precipitation best of all.

TABLE XVIII.

Present.	Recovered.	Percentage.
0.02 grm.	0.0196	97.8
0.03 "	0.0294	98.0
0.05 "	0.0489	97.8
0.10 "	0.0986	98.6
0.20 "	0.1956	97.8

Copper in Steel.

When estimating copper in steel and such iron compounds as contain less than half per cent, it is necessary to weigh off from 5 to 10 grms. of the sample. Such large amounts of iron could not be precipitated as basic acetate without involving a very cumbersome volume of liquid.

The procedure in such cases (and in many others which will suggest themselves) so as to make use of the soda cyanide titration is as follows:—

Dissolve the sample in dilute sulphuric acid, and precipitate as sulphide with sulphuretted hydrogen, or as subsulphide with soda hyposulphite. The latter is perhaps the more convenient for occasional use, and for the

sake of clearness we will describe its processes beyond the point at which they diverge from the usual instructions.

It is very convenient to perform the preliminary operations in a flask. Having filtered the supernatant solution from the precipitated subsulphide, wash two or three times by decantation. The precipitate settles so readily that little more than traces of copper will have passed on to the small paper. Ignite it, replace the residue in the flask, and add a small quantity of nitro-hydrochloric acid—10 to 20 c.c. Heat to boiling, and add separately a few crystals of potassium chlorate. A large excess of hyposulphite should be avoided, else the decomposition at this stage becomes troublesome. When no solid matter remains save a few pieces of sulphur, cool, dilute, neutralise, make alkaline with soda carbonate, and titrate with standard cyanide as previously explained.

It is unnecessary to offer any array of figures in this connection. The separation is one already well established, and that the estimation may be made as proposed has been abundantly proved.

LABORATORY NOTES.

By H. JERVIS.

A CASUAL visit to many laboratories reveals the presence of pieces of apparatus which would be far less cumbersome if its composite pieces had been such as were known to be obtainable, but considered to be too expensive.

A common example of this is seen in the number of makeshifts for tubulated bottles and other articles requiring a hole here or there. This of itself would show that the ability to perforate glass is not largely possessed by the everyday operator.

The process is so serviceable that a few lines explaining the *modus operandi* will be acceptable to many who have never previously tried to do the job, and perhaps helpful to those who have previously left the matter to the "engineering shop," and finally concluded that it needed special apparatus. The operation really belongs to that great class of "tricks" practised by itinerant repairers and scientific amateurs.

Take a hole through a cover-glass as the simplest case. Make a few scratches in the form of an asterisk with the point of a broken three-square file dipped in turpentine. Rest the cover on a wooden block or piece of cork; then turn the sharp point of a similar file backwards and forwards on the mark, or use the point of a broken file fitted in a brace, and the hole is through in a few minutes. In some cases, usually with sheet glass, it is better to bore from each side.

Take now a Winchester. Say we need an aspirator or some form of H₂S apparatus, and want to make a hole $\frac{1}{2}$ inch diameter. Scratch on the asterisk. If only old files are available, use first the point of a three-square to start the hole. Then break the point off and work with new and larger point, and so on until the hole is $\frac{3}{8}$ or $\frac{1}{2}$ inch diameter. A smart tap will break the file as required, and the new edges are very able cutters. When the indentation is thus enlarged, one of the sharp corners of the broken file can be used to groove it towards the centre with almost as much facility as a bradawl could be used on an indent in hard wood. A few turns of the file clears off the ridges and leaves the indent much deeper. If the hole is much cupped and nearly through, a smart tap with a file tang invariably makes a clean hole: there must be no hesitation in giving the tap and no blundering brute force. The hole made, it can be readily widened by working with a tapering file. An ordinary flat file is as good as any other shape, perhaps better—it does not jam so readily. Porcelain can be similarly perforated.

Throughout these operations the tool should be moistened with turpentine, or turpentine and camphor,

and such articles as bottles can be conveniently fixed in position, resting on a partly opened heavy drawer. As an alternative the boring may be done under water in a wash basin or deep sink. In this latter way, too, it is possible to shape sheet glass with a pair of scissors, but one must make haste carefully.

To those whose while it is worth a set of bits made from old files will suggest itself. The Archimedean brace is a good variety for small work. For very small holes the bow brace used largely by watchmakers and crockery rivetters is an acquisition which can be readily made.

The breaking of thick glass combustion tubing is really an easy operation. Teachers often break it by heating a nicked portion between coils of wetted paper, and dealers by working on brown paper with string; twine soaked with alcohol is another means. I have found all to be inferior to the following. Make a decided scratch at one portion of the tube, and then complete the circle in a fainter line—not necessarily fainter. An iron rod ($\frac{1}{8}$ or $\frac{1}{4}$ inch) meanwhile pushed up the burner of a muffle will now have become hot; lay it on the deeper scratch until it approaches blackness, and then, if the fracture has not already occurred, touch the heated glass with the tip of a wetted finger. The fracture is in most cases as even as possible. Occasional irregularities are levelled up with the pliers. The effect is greater and the danger of fracture less if the pliers are used from the outer diameter of the tube.

ON THE ESTIMATION OF LIME, ALUMINA, AND IRON IN MINERAL PHOSPHATES.

By M. L. LINDET.

THE attention of agricultural chemists has often been drawn to the part played by alumina and oxide of iron in the retrogradation of superphosphates, and to the difficulties which attend the estimation of these two elements in commercial phosphates. The numerous methods which have been proposed to effect this estimation all require a certain amount of delicate manipulation, and many of them give but uncertain results; they have further been the subject of a critical examination by M. Lasne (*Bull. Soc. Chim.*, pp. 118, 148, 237, 1896), which enables me to dispense with any description of them or to point out their relative disadvantages. The estimation of lime is, as a rule, conducted in the liquors from which the iron and alumina have already been separated; its exactness therefore depends on the processes to which I have just referred.

The most generally used method for the estimation of phosphoric acid consists of precipitating as ammonio-magnesian phosphate, in the presence of a large excess of citrate of ammonia, which keeps the lime, alumina, oxides of iron, manganese, &c., in solution. To then separate these oxides from the filtrates it is necessary to destroy the citric acid, either by evaporation, which never takes place without bumping, and calcination of the residue,—and this is always a long operation,—or by oxidation of this residue with fuming nitric acid, or a mixture of nitrate and chloride of potassium. These oxidations are in general very incomplete, inasmuch as the iron and alumina remain, in spite of everything, in solution, in the presence of ammonia.

It occurred to me that one might, with advantage, and for the purpose of destroying this citric acid, use the beautiful reaction recently described by M. Villiers (*Comptes Rendus*, cxxiv., p. 1349), that is to say, the oxidation of organic matters by nitric acid in the presence of manganese. The reaction, as a matter of fact, can give and has given perfect results.

The operation should be conducted in the following manner:—The ammoniacal liquors, from which the ammonio-magnesian phosphate has been removed, are saturated with nitric acid, and 0.5 grm. of sulphate or nitrate of manganese, and about 50 c.c. of nitric acid for every 20 grms. of citric acid, are then added. This mixture placed in a flask is gently heated, and the attack proceeds during the evaporation; nitric acid is added every time the action decreases; this can be easily seen by removing the flame. When a fresh addition of acid no longer causes any evolution of gas, we may rest assured that there is no longer any nitric acid present, and that it can no longer prevent the precipitation of iron and alumina by ammonia. The precipitate is collected and re-dissolved, and separated by the ordinary methods.

Chloride of vanadium (dichloride of vanadyle, VaOCl_2) may with advantage be substituted for the salts of manganese. Its action is much more energetic, and 0.1 grm. suffices for the rapid oxidation of 20 grms. of citric acid. Hypo-vanadate of ammonium, precipitated at the same time as the iron and alumina, is insoluble under the conditions of the experiment, above all in the presence of ammonia in excess. Instead of trying to separate the iron and the alumina, we can subtract from the weight of the calcined precipitate the weight of oxide of vanadium added; it therefore suffices to make use of a 1 per cent solution of chloride of vanadium, and to take, for precipitating with ammonia, 10 c.c. of the liquid, in presence of a known weight of sesquioxide of iron.

Whether we use salts of manganese or salts of vanadium for the destruction of the citric acid, it is easy, in the liquors from which the iron and alumina have been eliminated, to estimate the lime in the ordinary manner, —*Journ. de Pharm. et de Chim.*, Series 6, vol. vi., No. 6.

THE CALCINATION OF CARBONATED MANGANIFEROUS MINERALS, AND THEIR ASSAY.*

By N. DEVISSE.

BEFORE commenting on the different metallurgical appliances for the calcination of carbonated manganiferous minerals, it is indispensable to point out the thermo-chemical properties, whose interpretation will largely contribute to the elucidation of divers phenomena, and to the determination of the best conditions to allow of the minimum consumption of fuel.

In the first place, the chemical composition of these minerals, when subject to ordinary atmospheric conditions, is in a state of unstable equilibrium, tending towards the formation of pyrolusite, the original pink mineral becoming black. Owing to this conversion, heaps of manganiferous ore have been known to so alter that the percentage of manganese increased from 46 to 52 in a few years.

Diallogite, under the influence of air and rain, behaves in a similar manner, while the rain waters—by dissolving the carbonate of lime and magnesia which are generally present—render the mass porous, enriching it considerably, as in the previous case.

This metamorphosis is further in harmony with the thermo-chemical laws of M. Berthelot, according to which for 1 ton of ore, at 45 per cent contents of Mn, there would be a disengagement of 63.47 calories, equivalent to the heat of combustion of 8.4 kilos. of coal, from which we may conclude that pure diallogite is a combustible material.

This being established, we can now approach the study of the calcination of carbonated manganiferous minerals.

The roasting of these minerals may be carried out

* Abridged from the *Revue Universelle des Mines et de la Metallurgie*, Series 3, vol. xxxix., August, 1897.

either in heaps, kilns, or ovens. The first may be passed over. In new installations the choice of apparatus depends on the quantity of silica present in the mineral; if it is low, kilns are the best to use, having at the same time one or two ovens for the calcination of lumps when their proportion in the ore to be treated is considerable. If the ore contains as much as 8 per cent of silica, it is better to use only ovens.

As soon as the ore attains the temperature necessary for the dissociation of the carbonic acid, this silica unites energetically with the protoxide of manganese, forming a silicate, agglomerating the ore in masses whose volume will frequently entirely upset the operation, the charge descending in the most unexpected manner. It is then necessary to empty the furnace and break up the hard lumps, in which good crystals of the silicate may be often found.

In using the kiln, the ore and the fuel charged at the top absorb the heat of the products of combustion as they ascend, while the air entering below is warmed at the expense of the already calcined material; on arriving at the intermediate zone the air gives up its oxygen, producing, in conjunction with the combustion of the fuel and of the protoxide of manganese, the heat which will be absorbed by the dissociation of the carbonic acid and in the upper zone of decarbonisation.

The calcined ore is drawn out from time to time, at regular intervals, stopping when it appears warm, and the space formed at the furnace mouth is filled with a fresh charge.

Although pure diallogite is a combustible mineral, we must not conclude that the carbonated manganiferous ores, when once alight, will continue roasting of themselves; there is none in nature sufficiently pure to effect this.

Being always in conjunction with the isomorphous carbonates of lime and magnesia, with a certain proportion of silica, it requires for its calcination such a quantity of heat that we must still have recourse to fuel, but in amounts smaller according to the efficiency of the furnace, which must allow of the easy combustion of the protoxide of manganese and the regular descent of the charge.

The combustion of the protoxide of manganese depends on the uniform distribution of a sufficient quantity of air; this is best obtained by means of a damper. The regular descent of the charge depends entirely on the shape and section of the kiln, ovoids, and cylindro-conical furnaces; widening towards the top should be absolutely rejected, the best shape being cylindro-conical in vertical section, slightly widening towards the base. This form allows of the uniform descent of the charge and the parallel ascent of the currents of gas.

The section of the outlet should not be too large, or there will always remain a quantity of ore which cannot be removed.

Among the well-known blast-furnaces used, the best would probably be the Ayresome (Cleveland), and it is to this class that we are indebted for the principle on which the above-mentioned one was constructed.

When the ore to be calcined is siliceous, or for the calcination of large lumps, we have already said that the kiln is unsuitable; in these cases it is best to make use of large parallel ovens, about 7 metres long by 1.5 metres wide, and about 8 metres high. The floor which carries the ore is formed of rows of bricks, with spaces in between of about 0.15 metre in width, forming an arch 0.35 metre high in front of each oven door. The oven being empty, we begin by charging it with the unroasted ore from the previous operation, to about 0.25 metre thickness, putting the largest lumps over the spaces between the bricks; we then spread the first layer of fuel, and alternate layers of fuel and raw ore up to 0.4 metre high. When the charge reaches the height of the door it is bricked up, and the fire started with wood under each arch. The charging continues day by day, taking care to gradually diminish

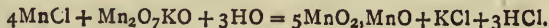
the quantity of fuel, especially for the last two layers, when finer ore must be used. As soon as the first charge becomes of a dull red heat, the fire below is allowed to go out, and the combustion proceeds from layer to layer. After four or five days the fire reaches the upper layers; when cool the door at the bottom is opened, and the mineral—which is in what is called the "liquid" state—runs out with ease. The operation lasts eight or nine days from beginning to end, and gives a return of about 400 tons per month.

The problem of calcining pulverulent minerals has not yet been satisfactorily solved; it cannot be done in a kiln or an oven as just described. It has been done in a "Pelatan" furnace, but at the expense of 12 to 13 per cent of coke.

The Assay of Ores.

Without reviewing the known methods of estimating manganese, we will simply point out a few facts deduced from the beautiful experiments made by Gorgeu (*Ann. de Phys. et de Chim.*, "On Manganous Acid," Series 3, vol. lvi., p. 153), the importance of which does not seem to have been sufficiently appreciated.

When we precipitate manganese in the presence of an oxidising agent, such as chlorine, bromine, &c., this metal tends to separate in the form of manganate, according to the formula $5\text{MnO}_2, \text{MO}$, MO being either protoxide of manganese, oxide of zinc, lime, &c. For example, when we add permanganate to chloride of manganese all the permanganate is decomposed; at the same time the liquid becomes acid, and a brown precipitate takes place. If we neutralise the acid set at liberty, as it is produced, we notice, after having added one equivalent of permanganate of potash to four of chloride of manganese, that three-quarters of the hydrochloric acid have been set at liberty, and that there is no longer any manganese remaining in solution:—



The presence of salts of lime, zinc, &c., thus casts suspicion on gravimetric estimations, while on the contrary it is necessary to the accuracy of volumetric methods, in which the object is to measure the quantity of oxygen absorbed by the manganese in its precipitation in the state of binoxide, or, rather of manganous acid. We strongly recommend the use of volumetric methods, which are of extreme accuracy whenever we add to the solutions a sufficient quantity of a soluble salt of zinc or lime; we even advise keeping a certain quantity of these bases in suspension at the moment of precipitation.

Under these conditions all the manganese will be precipitated in the state of manganous acid, in the manganites of zinc or lime which are formed; the manganite of manganese is not formed except when this metal is alone, as, for example, when we submit precipitated carbonate of manganese to the action of chlorine in excess.

Finally, in Volhard's method, the most elegant and rapid of all the volumetric methods, it is not necessary to transform the chlorides into sulphates, if we make it a rule to add precipitated oxide of zinc to the dilute neutral liquid to which the permanganate of potash is to be added. In fact, the hydrochloric acid set at liberty in the reaction—



is in this manner neutralised immediately on its formation.

Jubilee Medal.—The Queen has been graciously pleased to bestow upon Mr. Walter Hills, President of the Pharmaceutical Society of Great Britain, a mark of Royal favour in connection with the sixtieth anniversary of Her Majesty's reign, by presenting him with a medal to be worn as a decoration commemorative of that event.

ON THE COMPOSITION OF CERTAIN
CANADIAN VIRGIN SOILS.*By FRANK T. SHUTT, M.A. F.I.C., F.C.S.,
Chemist, Dominion Experimental Farms.

(Continued from p. 206).

North-West Territories and Manitoba.

THE prairie soils of the North-West Territories and Manitoba are justly noted for their productiveness. They contain, as a rule, large percentages of all the essential constituents, and are characterised by percentages of humus and nitrogen far above the average. The prevailing surface soil, speaking generally, is a black or greyish black loam in which the vegetable matter is well decomposed and thoroughly incorporated with the inorganic compounds of the soil. It varies in depth from a few inches to one, two, or even more feet, and over large areas is underlaid with a heavy clay subsoil.

Occasionally we have had sent to us soils from certain districts in the North-West Territories, in which it is stated that poor yields are obtained. On examination, these soils have been found to possess plant food in adequate quantities for crop requirements. Further, they have usually been found to be free from alkali. Investigation has shown that the trouble was, not in the lack of plant food, but rather in the climate; a scanty rainfall being really the cause of the poverty of growth. In districts subject to drought irrigation, if feasible, would render such soils most fertile. An illustration of this is afforded by the late irrigation trials at Calgary, which have proved so successful from an agricultural point of view. In this connection we have to add that unfortunately no means for extensive irrigation appear practicable for several of the districts here referred to in the North-West Territories.

The presence of "alkali" in the soil in patches over certain areas in Manitoba and the North-West Territories is intimately connected with the question of rainfall. An alkali area may be restricted to a few square feet, or it may cover some acres. Patches of alkali soil occur surrounded by land of great productiveness.

The formation and retention of alkali are dependent upon the amount of water the soil receives and the facility for subsoil drainage. We need not now discuss the occurrence of alkali nor its nature, but it is valuable to note that, though the amounts of alkali found in samples submitted to us are often so great as to render the growth of wheat impossible, we have invariably found such soils to be rich in mineral and organic constituents. This shows that the soil proper is capable of acting as a fertile one, provided the alkali were got rid of by drainage, irrigation, or treatment with gypsum.

In Table III. we have given analytical data of seven surface soils from the North-West Territories. Though there is a greater uniformity in the texture and composition of soils upon the prairies than among soils of the Eastern provinces, no claim is made that the vast extent of the Territories is represented by these samples—they are altogether too few in number. They may serve, however, to indicate the general character of the soils over certain large areas.

Without discussing these soils in detail, attention may be called to their high nitrogen content and the large amounts of organic matter that are almost invariably present. These soils also contain above the average amount of potash. Our results do not show them to be noted for phosphoric acid, though they possess quantities quite equal to those in many very fertile soils. The great depth of the surface soil over large areas accentuates our deductions respecting the vast stores of plant food laid up in the plains for future crops. We are of the belief that

where poor crops only are procurable the climatic conditions are rather at fault than that there is a lack of plant food. Even in soils containing injurious amounts of alkali we have found, as already pointed out, an abundance of fertilising ingredients; drainage, if there is an adequate rainfall, frequently being all that is necessary to bring them into a state of productiveness.

Soil No. 37 represents the unfertilised and uncropped prairie soil of the Red River Valley, Manitoba. It was taken from Section 31, Township 4, Range 1, West. The uniformity in the character of the soil over a very large area in Manitoba makes the data here presented of more than ordinary importance.

The surface soil, which is fairly uniform throughout its depth, averages a little over 2 feet in thickness and merges gradually into the subsoil, which is blue clay. The latter, as tested by boring for water at this spot, extends at least to a depth of 250 feet.

The soil is deep black loam, of a fine and peculiarly characteristic granular order. It reduces easily between the fingers in the air-dried condition to a greyish brown powder. Though there is present a considerable amount of undecomposed root-fibre, the soil proper exhibits a remarkable homogeneity, indicating a process of physical refining in its formation and a uniformity in the chemical composition. The very large amount of organic matter present is undoubtedly most intimately incorporated with the clay and sand which constitutes the basis of the soil.

Though containing a large amount of clay, laboratory experiments show that this soil does not readily "puddle" on moistening, nor on subsequent drying does it form into a hard mass, but readily granulates on slight pressure.

The large amount of organic matter present has already been remarked; it exceeds 25 per cent of the water-free soil. The nitrogen is found to be practically 1 per cent, which would show that there is contained in an acre of soil to the depth of 1 foot more than 30,000 pounds of this element. Since ordinary fertile soils to a like depth contain from 3500 to 10,000 lbs. of nitrogen per acre, the vast reserve of this valuable constituent in this prairie soil is apparent.

This soil is also very rich in potash, containing an amount far in excess of that ordinarily met with in fertile soils. But two other virgin soils examined by us approach its potash content, 1.03 per cent.

Of phosphoric acid it contains 0.29 per cent. This also is above the average, most of our good soils showing between 0.15 per cent and 0.25 per cent phosphoric acid.

We may safely conclude that there is here ample scientific proof of the well-nigh inexhaustible stores of plant food, and that this prairie land, as regards the elements of fertility, ranks with the richest of known soils.

Concerning the prairie soil of the Red River Valley, Dr. Geo. M. Dawson, Director of the Geological Survey of Canada, wrote some years ago as follows:—

"Of the alluvial prairie of the Red River much has already been said, and the uniform fertility of its soil cannot be exaggerated. The surface, for a depth of two to four feet, is a dark mould, composed of the same material as the subsoil, but mingled with much vegetable matter. Its dark colour is no doubt due in part to the gradual accumulation of the charred grasses left by the prairie fires. The soil may be said to be ready for the plough, and in turning the tough thick prairie sod, the first year a crop of potatoes may be put in, though it is not efficiently broken up till it has been subjected to a winter's frost. When the sod has rotted, the soil appears as a light friable mould, easily worked and most favourable for agriculture. The marly alluvium underlying the vegetable mould would, in most countries, be considered a soil of the best quality, and the fertility of the ground may, therefore, be considered as practically inexhaustible.

"The area of this lowest prairie has been approximately stated as 6900 square miles, but the whole is not at present suitable for agriculture. Small swamps are scattered pretty uniformly over its surface. The greater

* Read before the British Association (Section B), Toronto Meeting, 1897.

TABLE III.—Analyses of Soils (Water-free), North-West Territories and Manitoba.

No.	Locality.	Surface or subsoil.	Character of soil.	Potash.	Phosphoric acid.	Nitrogen.	Lime.	Loss on ignition (organic and volatile matter)
30.	Yorkton, N.W.T. . .	Surface.. ..	Black sandy loam ..	0'49	0'21	0'504	0'06	14'01
31.	" " " " " "	Subsoil.. ..	" " " " " "	0'42	0'09	0'130	0'75	8'18
32.	Saltcoats, " " "	Surface.. ..	" " " " " "	0'34	0'21	0'571	2'90	13'54
33.	Moosomin, " " "	" " " " " "	Black loam.. ..	0'36	0'11	0'479	0'95	11'79
34.	Calgary, " " "	" " " " " "	" " " " " "	0'44	0'17	0'447	0'92	12'23
35.	Tilley Township, N.W.T. " " "	" " " " " "	" " " " " "	0'27	0'18	0'398	0'37	11'13
36.	Vermillion Hills, N.W.T. " " "	" " " " " "	" " " " " "	0'17	0'17	0'354	0'50	10'43
37.	Red River Valley, Manitoba " " "	" " " " " "	" " " " " "	1'03	0'29	1'005	1'89	26'29

TABLE IV.—Analyses of Soils (Water-free), Ontario.

38.	Sinclair Township, Muskoka " " "	Surface.. ..	Sandy loam . . .	0'11	0'27	0'186	0'12	8'74
39.	Chaffey Township, Muskoka " " "	" " " " " "	" " " " " "	0'08	0'12	0'139	0'40	6'79
40.	Chaffey Township, Muskoka " " "	Subsoil.. ..	Sand " " " " "	0'08	0'18	0'074	0'20	3'53
41.	Franklin Township, Muskoka " " "	Surface.. ..	Light grey loam .	0'61	0'18	0'103	0'76	6'31
42.	Franklin Township, Muskoka " " "	Subsoil.. ..	" " " " " "	0'02	0'08	trace	0'66	3'70
43.	Perry Township, Muskoka " " "	Surface.. ..	Sandy loam . . .	0'04	0'18	0'296	0'08	9'40
44.	Perry Township, Muskoka " " "	Subsoil.. ..	" " " " " "	0'06	0'18	0'119	0'13	5'10
45.	Brunel Township, Muskoka " " "	Surface.. ..	Clay loam " " "	0'46	0'17	0'084	1'28	2'94
46.	Brunel Township, Muskoka " " "	Subsoil.. ..	" " " " " "	0'29	0'09	0'064	1'07	2'39

TABLE V.—Analyses of Soils (Water-free), Quebec.

47.	Arthabaska, Quebec	Surface.. ..	Sandy loam . . .	0'16	0'17	0'296	0'35	8'68
48.	" " " " " "	Subsoil.. ..	" " " " " "	0'17	0'18	0'184	0'29	5'46
49.	St. Adelaide de Fabos	Surface.. ..	Red sandy loam..	0'44	0'07	0'215	0'16	7'85
50.	Soulanges, Gaspé..	" " " " " "	Grey sandy loam ..	0'39	0'33	0'198	0'47	7'76
51.	" " " " " "	Subsoil.. ..	" " " " " "	0'47	0'30	0'049	0'73	3'67
52.	Lievre River, " " "	Surface.. ..	Clay loam " " "	0'11	0'19	0'179	1'23	5'77
53.	" " " " " "	Subsoil.. ..	" " " " " "	0'10	0'19	0'171	1'17	5'62
54.	Joilette, " " "	Surface.. ..	Black clay loam..	0'40	0'28	0'218	0'82	8'06
55.	" " " " " "	Subsoil.. ..	" " " " " "	0'44	0'29	0'030	1'05	2'09
56.	Bonaventure, " " "	Surface.. ..	Reddish yellow clay loam " " " " "	1'17	0'19	0'249	0'10	12'37

part of these swamps are, however, so situated as to be easily drained, either into the Red River or some of its tributaries, which are usually depressed 30 or 40 feet below the level of the surface.

"As a measure of the possible agricultural capacity of this great valley, take one-half of the entire area, or 3,400 square miles, equalling 2,176,000 acres, and for simplicity of calculation, let it be supposed to be sown entirely in wheat; then, at the rate of 17 bushels per acre, which, according to Prof. Thomas, is the average yield for Minnesota, the crop of the Red River Valley would amount to 40,992,000 bushels."

Ontario.

The review of soils in this province will be restricted to certain surface and subsoil samples collected in the district of Muskoka (Table IV.)—a district lying somewhat over 100 miles north of Toronto, and considered, for the most part, more picturesque than agricultural; it is rocky and abounding in lakes, well timbered—save where destructive fires have swept through,—with stretches of fairly good, though, as a rule, light soils along the river valleys and

on the tower levels. Our data respecting virgin soils in other parts of the province are too fragmentary to warrant their insertion in this paper.

Soil No. 38.—From Sinclair Township. A shallow very loose sandy soil; the subsoil of hard pan is found at a depth of from 6 to 12 inches. Though moderately rich in phosphoric acid, nitrogen, and humus, it is below the average in potash and lime.

Soils Nos. 39 and 40.—Surface and subsoil from Township of Chaffey. A shallow sandy loam running into a subsoil of sand. Hard pan exists at a depth of 15 inches. The surface soil is deficient in potash, but otherwise of medium quality as regards plant food.

Soils Nos. 41 and 42.—From Franklin Township. The surface soil is a light grey clay loam, high in potash, fair in phosphoric acid, and low in nitrogen; lime is present in an amount that might be considered large for Muskoka soils.

Soils Nos. 43 and 44.—Perry Township, Parry Sound district. Soil and subsoil. The country is described as level or gently sloping, with no rocky bluffs, and well timbered with excellent hardwood.

Both samples are light and sandy in character, and exceedingly low in potash and lime. Regarding the surface soil, we may say that the percentage of phosphoric acid is fair, and that in nitrogen it is above the average, compared with soils of this character in this district.

Soils Nos. 45 and 46.—Surface and Subsoil from Brunel Township. The surface soil is a clay loam of a light grey colour, from 8 to 12 inches in depth. It is a fairly strong and retentive soil, and in this respect differs from the preceding members of this series. The features in its favour are the comparatively high percentages of potash and lime. In nitrogen and humus, however, the soil is poor.

It is thus seen that the soils of this northern part of Ontario are characterised by a preponderance of sand, the larger number being such as would be classed as light or very light loams. They are loose in texture and very apt to dry out in season of drought. Though scarcely heavy enough for wheat, they grow good crops of oats and potatoes. Being responsive to manures, large yields of root and fodder crops can, under good system of culture, readily be obtained in favourable seasons. The district is better adapted for grazing and dairying than for the growth of cereals.

Quebec.

Table V. presents the data obtained from the examination of ten soils from the province of Quebec. They, as the preceding samples, have been selected as typical average soils; not, on the one hand, representing the richest, nor, on the other, the poorest, lands.

Soil No. 47.—Surface soil from Arthabaska county. A sandy loam of fair quality; nitrogen and organic matter are present in quantities somewhat above the average, but the soil ranks rather low as regards mineral constituents.

Soil No. 48.—Subsoil to the above, and very similar in its proportion of potash and phosphoric acid. For a subsoil it may be considered high in nitrogen.

Soil No. 49.—A surface soil from Gaspé. It is a red sandy loam, containing fair quantities of potash and nitrogen, but low in phosphoric acid and lime.

Soil No. 50.—A dark grey sandy loam from Soulanges county. A light, warm, responsive soil. In all the elements of plant food it may be placed with soils of good average fertility.

Soil No. 51.—Subsoil to the above, in which the mineral elements are present in fair amounts.

Soil No. 52.—A heavy clay loam from the valley of the Lièvre River, Ottawa county. A strong retentive soil. With drainage it should be well adapted to the growth of cereals. Though low in potash for a clay soil, it may be regarded as of average fertility. Drainage and the application of lime have vastly improved its productiveness.

Soil No. 53.—Subsoil to above, and very similar to it, both chemically and physically.

Soil No. 54.—A clay loam from Joliette county; greyish black in colour, compact, and cohesive. Both in mineral constituents and nitrogen this soil is above the average. An application of 20 bushels of lime per acre, however, resulted in almost doubling the yield.

Soil No. 55.—Subsoil to No. 54. Stiff clay, grey to reddish brown.

Soil No. 56.—A surface soil from the county of Bonaventure. A reddish yellow loam, containing a slight preponderance of sand. The large amount of iron present masks the presence of the organic matter, of which there is a notably high percentage. Not unfrequently—indeed, one may say usually—a rough estimate of the organic matter—and, incidentally, nitrogen—present can be made from the colour of the air-dried soil. In soils, however, such as the one under discussion, containing high percentages of iron, the colour can no longer be used as a criterion of the soils richness in these constituents.

Much variation, as might be expected, in character and composition is to be observed among these soils. Though some possess but small amounts of certain constituents,

indicating inadequate quantities for the best returns, yet none fall below the limits of fertility previously discussed, and many are seen to compare most favourably with soils of recognised productiveness.

(To be continued).

EARLY AMERICAN CHEMICAL SOCIETIES.*

By Prof. H. CARRINGTON BOLTON.

THREE chemical societies were organised in the United States before the close of the first quarter of this century; one as early as 1792, the second in 1811, and the third in 1821. These societies were short-lived, local in jurisdiction, and without much influence on the progress of the science; but it is interesting to note that professional, teaching, and amateur chemists in America formed associations for mutual improvement, and for the advancement of their calling, long before their European brethren. The Chemical Society of London, the oldest in Europe, was founded in 1841, forty-nine years after the first American society; that of Paris dates from 1858, and that of Germany from 1868. American chemists were not impelled to form independent societies, owing to a lack of organisations for men of science, but they early felt the advantages of a specialised association. The society of 1792, and that of 1811, were both founded in a city honoured by the presence of the venerable and dignified American Philosophical Society, established by Benjamin Franklin in 1743.

The existence of these societies has long been known, but only through casual references to them by writers on the beginnings of science in the United States; Prof. Benj. Silliman, in his essay on "American Contributions to Chemistry," read at the centennial celebration of the discovery of oxygen, held at Northumberland in 1874, alludes to them incidentally; and Dr. Brown Goode, in his historical address to the Biological Society of Washington, barely mentions them.

The publications too, of the earlier societies, are very little known, being rarely found in the best libraries.

Under these circumstances it has seemed not altogether useless to summarise what information concerning these societies I have been able to gather, and to offer it as a contribution to the history of chemistry in the United States.

The three societies are:—

I. The Chemical Society of Philadelphia, founded in 1792.

II. The Columbian Chemical Society of Philadelphia, founded in 1811.

III. The Delaware Chemical and Geological Society, founded in 1821.

I. The Chemical Society of Philadelphia.

The Chemical Society of Philadelphia was undoubtedly the earliest organised body of chemists in either hemisphere, having been "instituted" in 1792. The society does not seem to have published records of its meetings, nor of the papers presented thereat, and, since at that early day the primitive local newspapers paid little attention to items of scientific interest, information concerning it is not readily obtained. I find, however, that it was flourishing in 1801-2, when it had the following officers:—

President—Dr. James Woodhouse.

Vice-Presidents—Felix Pascalis and John Redman.

Librarian—William S. Jacobs.

Curators—William Brown and John S. Dorsey.

Treasurer—John Y. Bryant.

Secretary—Thomas Brown.

The society held stated meetings each week.

The President of the Society, Dr. James Woodhouse

* Read before the Washington Chemical Society, April 8, 1897. From the *Journal of the American Chemical Society*, August, 1897.

(1770—1809), was at the time professor of chemistry in the medical department of the University of Pennsylvania, of which he was a graduate.

This chair had been held by Dr. James Hutchinson, and on his death, in 1793, Dr. Joseph Priestley, who arrived from England a few months later, was invited to succeed him, but he declined, preferring the quiet life of Northumberland, and Dr. Woodhouse was chosen instead. Dr. Woodhouse contributed several medical papers to the New York Medical Repository, and to Cox's Medical Museum; he also edited Chaptal's "Elements of Chemistry" (fourth edition, 1807, 2 vols.), and Parkinson's "Chemical Pocket-Book" (1802). He is said to have been the first to prove by comparative experiments the superiority of anthracite coal from Pennsylvania over bituminous coal from Virginia for intensity and regularity of heating power (Silliman).

The first vice-president, Felix Pascalis Ouvrière (1750-1840), had an interesting career. He was born in France, where he received his medical education; he emigrated to Santo Domingo, and while practising medicine there acquired an extensive knowledge of botany and other branches of natural history. In 1793 a revolt among the negroes compelled Pascalis to flee, and he took refuge in the United States, first at Philadelphia, and later at New York, where he resided for more than thirty years. He was the founder of the Linnean Society of New York, and the author of several medical papers and reports.

The second vice-president, Dr. John Redman, (1722-1808), was a native of Philadelphia, and educated in European medical schools and hospitals. In 1786 he was made president of the Philadelphia College of Physicians. He was regarded as one of the foremost practitioners of medicine of Philadelphia, but his methods now appear super-heroic.

Dr. John Syng Dorsey (1783-1818), one of the curators, was professor of surgery and afterwards of *matéria medica* in the University of Pennsylvania. He had a high reputation as a surgeon, but his qualifications for membership in a chemical society seem to have been based chiefly on the fact that in his youth he had attended the chemical lectures of Sir Humphry Davy (1803).

I have not found the roll of members of this early society, but it appears that Priestley, Hare, and Seybert were active in it. The ambition of the members is shown by the circumstance that in 1802 there was a standing committee prepared to "annalize every mineral production" brought before them, and to give "an accurate account of each specimen free of expense."

The meeting held October 24, 1801, was made memorable by the appointment of a committee for the "discovery of means by which a greater concentration of heat might be obtained for chemical purposes." On this committee was placed among others Robert Hare, then only twenty years of age; but so soon as December 10th of the same year he reported to the society, on behalf of the committee, his invention of the "hydrostatic" (oxy-hydrogen) blowpipe. I need not here eulogise this important and useful invention, which yielded such a fruitful harvest of discoveries. This alone justified the existence of the first of chemical societies. In the following year the society caused Dr. Hare's account of this blowpipe to be printed in a pamphlet of thirty-four pages, 12mo., with one plate. This now rare booklet bears the title "Memoir on the Supply and Application of the Blowpipe, containing an account of a new method of supplying the blowpipe either with common air or oxygen gas; and also of the effects of the intense heat produced by the combustion of the hydrogen and oxygen gases. Illustrated by engravings. Published by order of the Chemical Society of Philadelphia, to whom it was presented by Robert Hare, jun., Corresponding Member of the Society. Philadelphia. Printed for the Chemical Society by H. Maxwell, Columbia House, 1802."*

Robert Hare's subsequent career as professor of chemistry in the medical school of the University of Pennsylvania from 1818 to 1847, is well known and accessible to all enquirers.

How much longer this association of chemists continued to meet, I have not ascertained. But the work of this society was evidently remembered by those who, ten years later, founded a new one, inasmuch as they designated it by the prefix "Columbian" to avoid confusion.

(To be continued).

THE PRODUCTION OF HALOIDS FROM PURE MATERIALS.*

THIS Report consists mainly of an abstract of portions of a paper lately published by the Secretary in the *Journal of the Chemical Society*.

It is well known that many chemical changes depend upon the presence of water among the acting substances, and some chemists have been tempted to suspect that possibly chemical change may not occur at all in the absence of moisture. On the other hand, there remain a very substantial number of changes which have not hitherto been "stopped" by the careful withdrawal of water from the sphere of action. Among these are the forming of ozone from oxygen, and the combining of certain metals (for example, mercury) with chlorine.

For some time past members of the Committee have been occupied in re-examining some of these latter phenomena. Great care was taken in preparing the various materials required; and novel and stringent methods of drying them were employed. Advantage has been taken of the opportunities which occurred during the progress of the work to re-examine the influence of sunlight, and of the silent electric discharge on highly purified chlorine.

The following is a summary of the results obtained:—

1. Carefully purified specimens of mercury, made by three distinct methods, were found to combine rapidly and completely with carefully dried chlorine.
2. Carefully purified mercury was also found to combine rapidly and completely with well-tried bromine and iodine.
3. Chlorine prepared by the electrolysis of silver chloride and dried by a brief exposure to phosphoric anhydride is not condensed when submitted to the action of the silent electric discharge.
4. Chlorine from the same source (see 3) becomes more sensitive than before to the action of sunlight, after the addition of a trace of damp air.
5. Lead glass, which is readily corroded when heated in damp chlorine, is unaffected by the same gas after it has been well dried.

As bearing on the general question, it may be mentioned that it was shown in the original memoir (*loc. cit.*) that well-dried ozone undergoes spontaneous decomposition far more rapidly than the damp gas. That is to say, the reaction $2O_3 = 3O_2$ is retarded, and not facilitated, by the presence of water. It remains to be seen if other and analogous reactions, some of which will shortly be investigated, are effected in a similar manner.

Contribution to the Biological History of the Phosphates.—L. Joly.—The solution of ammonium molybdate in dilute nitric acid so often employed in analytical chemistry for the detection of phosphoric acid serves us to detect its presence in animal tissues.—*Comptes Rendus*, cxxv., No. 15.

* The substance of a Report of a Committee consisting of Professors Armstrong and Dunstan, and Messrs. J. D. Cundall, C. H. Bothamley, and W. A. Shenstone (Secretary). Read before the British Association (Section B), Toronto Meeting, 1897.

* A copy of this is found in the Army Medical Library, Washington, D.C.

NOTICES OF BOOKS.

The True Chemistry. ("Chimie Vraie"). The Rigorous Application of Two General Laws of Chemical Action. By E. J. MAUMENÉ, Dr. es Sc. Pp. 205. Paris: P. Vicq-Dunod and Co. 1897.

FOR thirty-seven years M. Maumené has been working on his theory, and claims to have given thousands of proofs of the accuracy of the two laws discovered by him, viz., the "Law of Contact" and the "Law of Mixture." In a previous work, "A Treatise on the General Theory of Chemical Action" (1880), the author admits that some of his calculations were only approximate, and that this might have prejudiced some persons against them; but in this last volume, just published, every calculation is exact, simple, and uniform, and can be followed by any one. These two laws, he claims, will greatly simplify the theory of chemistry, by superseding it, through their simplicity. Basicity, affinity, dissociation—all can be shown, and worked out in plain figures, by one or the other of M. Maumené's two fundamental laws, which make "The True Chemistry."

The essential aim of chemistry is to know exactly what takes place in any change, so that under given circumstances the result of any other reaction can be foretold; this we can already do to a certain extent, but there is generally *something* which does not fit; it may be called the exception which proves the rule, but it may also be said that it is the exception which proves the rule to be wrong. If by calculation all the unknown reactions can be predicted, then it must be admitted that there is much to say in favour of M. Maumené's theory. It must, however, be left for the chemical world at large to judge; true philosophers will always accept new ideas, however startling, when once they are proved beyond any doubt to be correct.

This volume gives ample evidence of an enormous amount of trouble and thought, spread over many years, and the author deserves the thanks of all chemists for the ideas he has given them; it also stands before many other works hailing from across the channel,—it has an excellent index.

The Induction Coil in Practical Work, including Röntgen X Rays. By LEWIS WRIGHT. London: Macmillan and Co.

IN view of the considerable interest that has recently been directed to electrical discharge in high vacua by Dr. Röntgen's discovery involving the use of powerful and costly induction coils, the book before us supplies a want, and is likely to be greatly appreciated. The author in his preface explains that his object has been to give practical help to the efficient and safe use of an induction coil; and this idea seems to have been faithfully adhered to throughout the book.

In the first chapter, of twenty-two pages, the simple principles of electrical induction are lightly touched upon,—the woodcuts, by the way, strike us as old acquaintances,—but the matter is fresh and quite up to date.

In Chapter II., after briefly describing the early Du Bois-Reymond apparatus, the modern coil is thoroughly explained, diagrams and drawings being given of the various parts, not as a guide to amateur coil-makers, but simply to give the general principle upon which the modern coil is built.

In Chapter III. the instructions for the "care of a coil" and the use of primary batteries are well worth noting by any who are using this class of apparatus for the first time, for a little neglect will easily ruin a most expensive coil, and by simple attention to the details noted both coil and battery will be kept in good order.

A short chapter is devoted to "Miscellaneous Experiments," probably to whet the appetite of the beginner,

and then three chapters are given to the electrical discharge in rarefied gases; brief accounts are given of De la Rive's experiments, Plücker's researches on stratified discharges, and others.

Chapter VII., on "The Discharge in High Vacua," is almost exclusively devoted to the researches of Sir W. Crookes, whose papers on Molecular Physics and Radiant Matter are extensively quoted and illustrated.

The remaining portion of the book is occupied by a discussion of Professor Röntgen's discoveries and their developments. Lenard's early work on the cathode rays is noted, and then the now old story of the X rays is retold. Some good practical advice is given as to the use of the Crookes tube in practice, which will repay the attention of any one using this expensive and easily damaged apparatus. It is easy to see that the author is writing from practical experience, and his instructions will be found of great value.

There is a very "made in England" style about the book, and the author shows a great—and one is inclined to think almost too great—contempt for Continental apparatus.

A footnote at the end of the book refers to a new form of contact-breaker recently described, in which the chief novelty lies in the fact that the swinging hammer of the break has to swing some distance before the contact is broken, thus making a very sudden break, causing less sparking and more inductive effect. It does not seem to be generally recognised that this principle is embraced in the very simple and primitive form generally used on Continental coils, where the mass of iron forming the hammer is fixed on the end of a somewhat long flexible spring, the platinum contact being placed some inch or so lower than the iron head.

The book is well printed and plenty of illustrations are given, making it both interesting and instructive.

Report of the Council of the Institute of Mines and Forests on the Gold and Forest Industries of British Guiana for the Year ending June 30, 1897. Demerara: W. H. Hinds, Echo Office, 1897.

IT is satisfactory to note that the gold industry of British Guiana shows signs of improvement, the output for the year ending June 30, 1897, being 128,333 ounces, as against 119,422 ounces in the previous year. It, however, still falls short of the outputs for 1892-3, 1893-4, and 1894-5, which were respectively 138,279 ounces, 137,822 ounces, and 128,760 ounces.

The past year has been marked by the first returns of any importance from quartz crushing, the Barima mine having produced over 7000 ounces.

Of the five districts into which the gold-bearing territory is divided, the most encouraging increase of all is to be found in the Potaro District; it has shown a steady increase since 1892, and now heads the list with 30,434 ounces, and it is hoped that with the extra facilities afforded by the Demerara-Essequibo Railway and the Government Road that the next twelve months will see a still further increase in the prosperity of this important district.

The number of labourers employed in the gold mining industry throughout the colony on June 30, 1897, was registered as 17,050.

The Governor has placed the services of Prof. Harrison, the Government analyst, at the disposal of the Institute for the examination of minerals free of cost, on condition that the samples are of public interest and that the results are published for general information.

The exports of forest products, including timber, shingles, charcoal, &c., also show a most encouraging increase over last year, especially viewed with regard to the prevailing depression in the staple product—sugar. The export of timber alone has almost doubled itself,

being for the year ending June 30, 1897, 404,234 cubic feet, valued at over 148,000 dollars.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxv., No. 15, October 11, 1897.

Further Experiments on the Liquefaction of Fluorine.—H. Moissan and J. Dewar.—Already inserted.

Direct Transformation of Heat into Electric Energy.—Marcus Deprez.

Spectra of the Coloured Components of Double Stars.—Sir William Huggins.

Spectra of the Principal Stars of the Trapezium of the Nebula of Orion.—Sir William Huggins.

New Mixed Platinous Salt.—M. Vézes.—The limited action of the hydrochloric, hydrobromic, or hydriodic acids upon potassium platinonitrate, $\text{Pt}(\text{NO}_2)_4\text{K}_2$, gives rise to the elimination of half the nitrous acid contained in this salt, and to the formation of mixed chloro-, bromo-, or iodo-nitro-salts very stable in aqueous solution; the platino-dichloro-nitrite, $\text{PtI}_2(\text{NO}_2)_2\text{K}_2$; lastly, the platino-diiodo-nitrite already obtained by Nilson. Oxalic acid gives rise to an analogous reaction with the sole difference that, by reason of its basicity, a single molecule of acid in place of two intervenes in the reaction. The properties of potassium platino-oxalonitrite are capable of being utilised in the separation of the platinum metals.

Procedure of Separating and Distilling Bromine from a Mixture of Alkaline Chloride and Bromide.—H. Baubigny and P. Rivals.

Reversible Transformation of Styrolene-meta-styrolene under the Influence of Heat.—G. Lemoine.—The reversible transformation of styrolene into meta-styrolene under the influence of heat recalls by its general course that of phosphorus, cyanogen, and cyanic acid; it tends progressively towards a limit expressed by a tension of the vapour of styrolene.

On Two Coloured Reactions of Pyruvic Acid.—L. Simon.—Pyruvic acid, with the addition of potassa and then of sodium nitroprusside, yields a beautiful and intense violet-red colouration. This is a reaction which appears to belong to all the fatty amines; it has been verified for the three methylamines, for mono- and diethylamine, amylamine, and benzylamine; with the last-mentioned base the colouration is that of lye of wine.

Action of Nitric Acid upon Potassium Cobaltcyanide.—E. Fleurent.—A note which the author merely gives to "take date."

Bulletin de la Société de Pharmacie de Bordeaux.
September, 1897.

Colorimetric Estimation of Manganese.—M. Lemaire.—Manganese is found in small quantities in a large number of plants, and many botanists consider it necessary to their proper nourishment. The ordinary gravimetric and volumetric methods of analysis are not suitable for estimating these small quantities; but, on the other hand, the colorimetric method is very convenient. It depends on the reaction pointed out by Hoppe-Seyler: If, to a substance free from chlorine, and containing manganese, we add binocide of lead and nitric acid, we obtain, on heating to boiling, a violet colouration due to the formation of permanganic acid. This reaction is very sensitive, and will detect $1/2,000,000$ th part of man-

ganese. The leaves of a sample of wild chicory, dried at 100° , gave 19.20 per cent of ash and 0.0004 per cent of manganese. The roots of another sample contained 0.0002 per cent of this element.

The Agricultural Journal of the Cape of Good Hope,
Vol. xi., No. 5.

Rinderpest Conference.—At the conference held on August 19th on this important subject, the several methods which have been employed for immunising cattle were discussed. It was at first expected that Dr. Koch's method of inoculating the healthy animals with the bile of a diseased one would confer immunisation on them, but there has since been a considerable difference of opinion as to its value. Unfortunately we are not in possession of a simple practical test which will enable us to distinguish between a safe bile, permanent in its effects, and biles which are either too strong or too weak. Even with biles which were to all appearances of standard quality, very varying results were obtained; and, further, it was found that the disease itself could be communicated in this manner. It is this uncertainty which is the most disappointing part of Koch's bile inoculation. Immunity has lasted as long as three or four months, but then the effect seems to wear off. As bile did not give the modified form of disease, it was found to be necessary to inoculate afterwards with virulent blood, and the present experience seems to be that Edington's method of inoculating a second time with virulent blood gives very good results.

Revue de Chimie Analytique.
Vol. v., No. 18.

Calibration of Graduated Glass Vessels.—M. Van-devyver.—The author thinks that the method recommended by M. Démichel, for correcting the error due to surface tension when calibrating glass vessels, is insufficient, as it assumes that the surface tension is constant and invariable, whereas such is not the case. For example, in gradually filling a glass with water, one-quarter at a time, the surface tension varies as follows:— $\frac{1}{4}$ full 6.35, $\frac{2}{4}$ full 5.22, $\frac{3}{4}$ full 5.09, full but not overflowing 4.89, full and overflowing 7.32. The author considers the following the best method to adopt for getting accurate results:—The vessel must first be cleaned with the greatest care, by washing it in acetic acid, caustic potash, then ether, alcohol, and finally rinsing several times with distilled water. It is then dried by inspiration, taking care to keep out dust by lightly plugging with cotton-wool, through which the glass tube passes; when dry, distilled water is run in up to the mark, and the operation proceeded with in the ordinary manner: by this plan the possible error due to the variation of the surface tension is reduced to a minimum.

Estimation of Copper as Iodide.—M. Willenz.—Will be inserted in full.

Calibration of Flasks by Weighing.—A. Démichel.—The author gives a number of equations and formulæ for calibrating flasks, which he claims will considerably simplify the operation.

A Tabular Atlas of the Chemistry of the Metals is being prepared by Mr. John Freemont Sleeper. It comprises the arrangement on a novel plan in sectional and tabular form, devised to rapidly convey information of the data relating to the history, distribution, properties, &c., of the metals of the alkalies, cæsium, rubidium, and potassium. The author has been preparing this work for the past ten years, and hopes to have it published before long.

MISCELLANEOUS.

The Goldsmiths' Institute, New Cross.—A course of 25 lectures on "Coal Tar Distillation," to be given on Wednesday evenings at 8.30 p.m., by Mr. W. J. Pope, was commenced on the 27th inst. The course comprises the determination of boiling-points, the latent heat of vapours, specific heat of liquids; the composition and valuation of tars; plant and methods used: separation and purification of benzene, toluene, xylene, &c.; preparation of anthracene, carbazole, pyridene, &c., &c. Special attention will be paid to methods of analysis and to the plant used in this country and abroad.

Salicylic Acid and Calcium Sulphite as Preservatives of Cider.—Salicylic acid was unanimously condemned in 1882 as a preservative of beer, and very little can be inferred as to the physiological effects of its continuous use. Kolbe took a daily dose of it for over a year, increasing from 0.5 gm. to 1.5 gm., without any noticeable effect. On the other hand, a case is recorded where 48 grains caused death in forty hours. Salicylic acid is easily detected; the sensitiveness of the ferric chloride test has been put as high as 1 part in 100,000. Dr. A. B. Griffiths found that a 1/5000th solution of salicylic acid had no action on the true alcoholic ferment. Messrs. E. Bailey and Chas. Palmer have made a series of experiments with various strengths of salicylic acid on cider. Six flasks of cider were used containing salicylic acid of the following strengths:—1/20,000th, 1/10,000th, 1/5000th, 1/1000th, 1/500th, and a blank. Distillations were made after twenty-four hours, seventy-two hours, eight days, twenty-four days, and fifty-two days. Though the effect of the preservative is not very marked till a 1/1000th solution is used, yet it does seem that a 1/5000th solution has a noticeable effect upon the alcoholic ferments, and the micoderma seems to do very well in a 1/1000th solution. The action of sulphurous acid and its salts as preservatives has not been studied to the same extent, though it is known to be an active germicide. Its detection in small quantities is difficult, but in quantities sufficient to exercise any preservative action, sulphurous acid may be readily detected by zinc and HCl. Experiments on the effects of various amounts of calcium sulphite on cider show that the action is retarding only, for a considerable amount of alcohol is produced after the fifty-second day, even in a 1/250th solution.

Histological and Chemical Study of the Action of Antiseptics on Muscular Fibres.—A. Riche.—Continuing his paper from the last number, the author describes further experiments on the albumen and juice of meat, and he comes to the conclusion that sulphurous acid and the bisulphites—notably that of lime—alter the normal structure of the meat, and that the muscular fibre does not remain intact under their action, even at ordinary temperatures; that the soluble albumenoid matters do not behave in the same manner as in the presence of water, even when subjected to a temperature below 100°, or even at 50°. For these and other reasons it is not to antiseptics, but to refrigeration, that we must look to solve the problem of the preservation of meat.—*Journal de Pharmacie et Chimie*, Series 6, vol. vi., No. 5.

On a New Alkaloid.—MM. Battandier and T. Malosse.—The authors have extracted a perfectly definite alkaloid from the young roots and from the bark of the *Retama sphaerocarpa*; they have given it the name *Retamine*. One kilogram. of the plant gave 4 grms. of the alkaloid. A number of its properties are here given. It melts at 162°; it possesses extremely energetic reducing properties, &c.; chloride of gold and phospho-molybdic acid are instantly reduced, but salts of silver and ferricyanide of potassium more slowly. The salts of retamine crystallise very easily and with great sharpness, with the exception of the nitrate, which has up till now only been

obtained as a varnish. The average of eight analyses comparing the carbon and hydrogen, and of twelve of the nitrogen, give it the formula $C_{15}H_{26}N_2O$. Retamine is therefore an oxy-sparteine, but different to any other one known.—*J. de Pharm. et Chim.*, Series 6, vol. vi., No. 6.

MEETINGS FOR THE WEEK.

- MONDAY, Nov. 1st.**—Royal Institution, 5. General Monthly Meeting. Society of Chemical Industry, 8. "The Adulteration of Portland Cement," by W. H. Stanger, M.I.C.E., and Bertram Blount, F.I.C. "An Improved Adjustable Drip-proof Bunsen," by W. P. Evans, M.A., Ph.D.
- WEDNESDAY, 3rd.**—Society of Public Analysts, 8. "Estimation of Acetates in the presence of Inorganic Salts," by Bertram Blount. "Estimation of Carbonic Acid in Natural Waters," by C. A. Seyler, B.Sc. "Detection of Gelatin in Cream," "A New Milk Preservative," "A New Milk Adulterant," by A. W. Stokes. "Note on the Graduation of Leffman-Beam Bottles," by G. E. Scott Smith and A. B. Searle. "An Improved Milk Scale," by H. Droop Richmond.
- THURSDAY, 4th.**—Chemical, 8. "Properties of Liquid Fluorine," by Professors H. Moissan and J. Dewar. "Liquefaction of Air and the Detection of Impurities," "Absorption of Hydrogen by Palladium at High Temperatures and Pressures," by Prof. Dewar.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1989.

DISTRIBUTION OF TITANIC OXIDE UPON THE SURFACE OF THE EARTH.*

By F. P. DUNNINGTON, F.C.S.

So far as I know, the discovery of the universal distribution of titanic oxide in the soil over the surface of the earth originated in the analysis of a cinder-like mass found about four miles from the University of Virginia and about a mile from "Monticello," the house of Thos. Jefferson being upon the slope of the same mountain.

This mass proved to contain 5·4 per cent of titanic oxide, and subsequently, upon analysis, to be of identical composition with the soil upon which it was found. The most probable suggestion of its formation is that it is a result of lightning. Other soil in that neighbourhood was examined for titanic acid, in all of which it was found; and subsequently, as many as eighty specimens of soil from various quarters of the globe. An account of these results was published by the author in the *American Journal of Science* for December, 1891.

It may be well here to give the averages for certain of these determinations:—

Titanic Acid in Soils. Averages of Determinations made.

	Percentage.
United States (40)	0·77
Great Britain (7)	0·75
Europe (10)	0·42
Igneous rocks—	
Germany (8)	0·56
Asia (8)	0·71
Japan (2)	0·55
Total average	0·63
Sandwich Islands (5)	3·17

It appears strange that any body existing in so considerable an amount in the soil should have been so constantly overlooked in the numerous analyses of soils that have been made; but this is in part to be accounted for by the fact that, for the purposes for which soil analyses are usually made, titanic oxide is considered as acting like alumina, and a distinction between them is regarded as a matter of little import.

This oversight is no doubt in part also due to the somewhat troublesome and unsatisfactory nature of the process of detecting and estimating this element which was employed prior to 1882, when Prof. A. Weller published in the *Berichte d. Chem. Gesell.* (xv., 2592) a method based upon the intense yellow colour of titanium trioxide, which is produced upon the addition of hydrogen peroxide to a solution of titanic sulphate.

Since publishing the foregoing results, I have been able to secure samples of soil from portions of the earth's surface not then represented; in these also the amounts of titanic oxide have been estimated as a percentage of the original soil and also of the ignited soil, which latter figures should be compared with what is found in igneous rocks. For a few of these determinations (which were made in duplicate) I am indebted to some of the students working in the Chemical Laboratory at the University of Virginia; those of the deep well borings were made by Mr. David Hancock, of Virginia.

- No. 81.—Red loam; and No. 82, dark grey loam; Brazil.
- „ 83.—Grey sandy loam, Liberia; per Mr. Apperson at the Chicago Exposition.
- „ 84.—Grey brown loam, St. Helena; per Professor Cleveland Abbe, Washington, D.C.
- „ 85.—Dark grey clay; and No. 86, light brown clay, Cerraloo, Mexico; per Mr. J. T. De Bell.
- „ 87.—A light brown clay; and No. 88, white clay, Sydney, Australia; per Prof. A. Liversidge.
- „ 89.—Brown-yellow loam, Launceston, Tasmania; and No. 90, brown-grey loam, Auckland, New Zealand; per Mr. H. J. Boyd.
- „ 91.—Light yellow clay, Araki, Japan; and No. 92, dark grey loam, Kawaja, Japan; per Mr. Apperson.
- „ 93.—Fine brown and green gravel, from earthquake eruption, Nayaya, Japan; per Rev. R. B. Grinnan.

(Nos. 94 to 104, from Dominion of Canada, were sent by Dr. G. M. Dawson, of Ottawa).

- No. 94.—Pale yellow fine sand; Champlain, Quebec.
- „ 95.—Brown loam; Ottawa.
- „ 96.—Grey clay; near St. Luis Dam, Ottawa.
- „ 97.—Brown loam; Beechwood, Ottawa.
- „ 98.—Pink loam; Murillo, Algomo.
- „ 99.—Black earth; Rosser, Manitoba.
- „ 100.—Fine black sand; MacGregor, Manitoba.
- „ 101.—Fine grey siliceous sand; Maple Creek, Assinboya.
- „ 102.—Dark grey sand; Canmore, Alberta.
- „ 103.—Light brown clay; Griffon Lake, B.C.
- „ 104.—Light red loam; Glacier, B.C.

(Nos. 105 to 114, drillings from the deep well at Wheeling, W. Va., obtained through Prof. Wm. Halleck, then at the Physical Laboratory of the U.S. Geological Survey).

The percentage of titanic acid found in these respectively is as follows:—

No.	Air-dried.	Ignited.	No.	Air-dried.	Ignited.
81.	1·33	1·79	93.	0·49	0·48
82.	0·74	0·83	94.	0·34	0·41
83.	0·62	0·66	95.	0·88	1·37
84.	2·33	3·00	96.	0·68	0·74
85.	0·22	0·25	97.	0·61	0·67
86.	0·14	0·20	98.	0·30	0·32
87.	0·69	0·79	99.	0·64	0·79
88.	0·43	0·48	100.	0·22	0·24
89.	0·50	0·61	101.	0·22	0·23
90.	1·40	1·57	102.	0·18	0·25
91.	0·52	0·55	103.	0·17	0·17
92.	0·43	0·46	104.	0·52	0·54

No.	From depth of—	Air-dried.	Ignited.
105.	275 feet	0·09	0·13
106.	500 „	0·79	0·89
107.	992 „	0·12	0·13
108.	1460 „	0·12	0·13
109.	2010 „	0·74	0·77
110.	2520 „	0·35	0·36
111.	3075 „	0·80	0·85
112.	3500 „	0·77	0·82
113.	4050 „	0·49	0·53
114.	4490 „	0·48	0·51

Average.. .. 0·475 0·512

These facts point plainly to the universal distribution of this element over the earth's crust. I would draw attention to the figure 0·56 per cent of titanic acid from the alluvial soil of the Yellow River in China, as determined in the former set of observations, as most probably presenting a fair average of the amount present upon the earth's surface.

* Read before the British Association (Section B), Toronto Meeting, 1897.

For all the above figures the total averages are 0.57 and 0.66. Omitting the Island of St. Helena, as exceptional, we have 0.515 and 0.588 respectively for the air-dried and ignited soils.

University of Virginia, August, 1897.

SEPARATIONS WITH ALKALINE ACETATES.

By HARRY BREARLEY.

(Concluded from p. 221).

VI. Zinc from Iron. General Considerations.

Zinc and manganese are the only two metals whose acetate separation from iron is so far favoured as to be commonly employed. In the case of Zn the fault is again the operators: carried out in the previously reiterated manner, the separation is one of the easiest and most complete of the series.

Two tests were made on solutions of 1 grm. iron and 0.1 grm. zinc. The zinc was estimated by precipitating as carbonate, and, so as to balance any errors, the value of the stock zinc solution was estimated under like conditions. The separations were carried out as before.

Acetate used.	Zinc recovered.
20 c.c.	0.1008 grm.
50 "	0.0976 "

No further observations on the separation of iron and zinc were made. If it be protested that such scant attention to zinc is by no means commensurate with its importance, it should be remembered that these papers are written from the standpoint of a Steel-works Analyst, who must make them as far as possible of momentary utility. In this light zinc is not an important element in combination with iron, and its separation therefrom is relatively of little value. It is by no means overlooked that in the larger world of analytical chemistry the separation is an important one, and therefore the tests were made with a view of allocating the element in the table of relative separability, if I may so term it, of the elements treated. There is, moreover, independent testimony to the claim of zinc to occupy its allotted place, in the fact that Mr. Jewett—whose paper I am unable to refer to—has found, in a somewhat different way, that manganese and zinc could be completely separated from iron by the acetate process, but not so nickel or cobalt.

The above defect is not really a very serious one. The influence of a particular variation can be readily made by anyone specially interested in the separation. It may be noticed, too, by those practically acquainted with the separation and estimation of the metals in Table XIX., that their relative behaviour with increasing acetate is a reliable indication of the liberties that may be taken with the processes without incurring the penalty of an imperfect separation.

And now, so far as I am aware, every metal whose acetate separation from iron is practised has been dealt with. There are, it is true, the alkaline earths; and as they are, more or less, always to be found in iron ores, slags, &c., they should not be omitted without reason. Good enough reason is to be found in the fact that the separation has long ago been established as a perfect one, no writer in standard books ever doubting its accuracy so far as to recommend a re-solution and precipitation of the basic ferric acetate.

A noteworthy point, not strictly within the scheme, is the behaviour of phosphoric acid. This compound invariably goes down with the iron in an ordinary acetate precipitation. Three tests were made to detect any possible variation from this course. Each test solution was made up of 1 grm. of iron and about $\frac{1}{2}$ a grm. of soda phosphate.

- I. contained total hydrate and 10 c.c. acetic acid,
- II. " " " " 30 " "
- III. " " no hydrate, and no free acid other than 30 c.c. acetic.

The iron was precipitated in each with minimum acetate. The filtrates were boiled to dryness; the residue of soda salts ignited, re-dissolved in dilute hydrochloric, neutralised with ammonia, acidified slightly with nitric acid, treated with the usual molybdate reagent, and placed side by side in a warm place. In I. there was only the faintest trace of a precipitate visible, and that chiefly where the rod had rubbed against the beaker. In II. there was somewhat more, but not enough to make any material difference to an analysis conducted in this way. In III. there was a very decided precipitate; such a quantity as makes a very serious difference when regarded as a deficiency. The amount was not determined quantitatively, it being rather the purpose of these remarks to guard against any possible error than to state definitely how great a percentage recovery is possible by these means.

The accompanying table (XIX.) affords perhaps the best general summary that could be given. It is only necessary to point out that each separation was made from a solution containing 1 grm. of iron and 0.1 grm. of the respective metal. Total hydrate was formed in the solution, and 10 c.c. acetic acid added. The volume of the solution was 1 litre. In some cases soda salts have been used, in others ammonia. This is the only difference in the treatment of the respective elements, and does not, I believe, involve in any case a change in the percentage recovery large enough to give any element precedence of its neighbour.

It was not practicable to include in Table XIX. the turbidity temperatures, although they afford the best proof I can give that the separations were made under comparable conditions.

The temperature at which a turbidity forms seems to have been generally overlooked. The observations are so readily made, and such reliable accompaniments of better or worse separations, according to their value and general conditions of working, that I venture to recommend the use of a thermometer for confirmatory purposes whenever a large number of estimations are made rapidly after the manner here set forth.

Wollcott Gibbs (CHEMICAL NEWS, xi., 102) has pointed out that the acetate should be added to the solution when cold, and then heated to boiling. This precaution has been repeated by Crookes ("Select Methods," p. 227, 3rd edit.), by Arnold ("Steel Works Analysis," p. 166), and others. Its advantage, when large excesses of acetate are used, is not questioned, otherwise the precaution becomes an unnecessary time absorber.

The fact that a perfect separation of Mn, Ni, &c., may be made by adding dilute acetate to the boiling solution, is one that in every-day practice has been regularly made use of. In dissolving the sample of (say) nickel steel, an approximately equal amount of acid is always used, and so one has some idea of the volume of alkali needed to effect neutralisation. Something less than this amount may be added at once, and then the neutralisation completed by adding in rapid succession several c.c. of the carbonate at a time. It is really unnecessary to wait for all traces of the preceding turbidity to disappear, because finally an addition of carbonate will cause a precipitate altogether unlike the preceding ones. Though hard to describe, the difference is easy to distinguish. The final precipitate is thicker, and pulls irregularly on the hand as the flask is whirled. In small volumes of solution it is almost like a jelly. This is an unmistakably permanent turbidity, produced almost as quickly as the description may be read, and not by any great excess of alkali either. To this turbid solution add 10 to 12 c.c. acetic acid. The turbidity quickly disappears, part of the acid going to form acetate. Dilute with hot water, heat fur-

TABLE XIX.
Percentage separation from iron of—

Dilute acetate. C.c.	Mn.	Zn.	Co.	Ni.	Cu.	Cr as CrO ₃ .	Cr as Cr ₂ O ₃ .	Al.
10	100.0	—	100.0	100.0	98.0	26.8	trace	Precipitation
20	100.0	100.8	99.0	99.0	92.9	30.8	(See p. 175).	of iron
50	100.0	97.6	97.5	95.2	69.6	38.8		not possible
100	100.0	—	93.8	90.0	53.1	47.5		(see p. 210).

SYNOPSIS.

Metal.	Acetate.	Oxide.
Manganese	Crystallises in plates; permanent in air..	Easily soluble in acids. Readily soluble in (NH ₄)Cl aq.
Zinc	Crystals may be sublimed as zinc acetate	Easily soluble in acids. Soluble in hot solutions of ammonium salts.
Cobalt	Crystallises; red needles	Easily soluble in acids; only slowly when cold. Soluble in hot NH ₄ Cl.
Nickel	Apple-green prisms	Insoluble in acetic acid (!) Only slightly soluble in hot NH ₄ Cl.
Copper.. . . .	Dark green crystals. There are also sesqui-, bi-, and tri-basic cupric acetates. These form when Cu is exposed in contact with acetic acid, as in common verdigris ..	Soluble in acids; slowly in NH ₄ salts. Soluble in boiling H ₂ O solutions of Al ₂ , Cr ₂ , Fe ₂ , with precipitation of oxides of the bases of these salts. Unacted upon by boiling solutions of Mn, Ni, Co, Zn.*
Chromic acid	The deficiency is due to an altogether different cause. The percentage recovery increases directly as the acetate. Acetic acid not notably advantageous.	
Aluminium	Decomposes on evaporation. There is a soluble basic acetate; but if this is heated or left to evaporate at ordinary temperature it deposits insoluble basic salts	

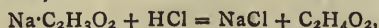
The relative separation of an element varies directly as the stability of the acetate and the solubility of the oxide under experimental conditions. The general behaviour of the acetates and oxides in the above synopsis is in accord with this statement.

* This division is exactly that Cu makes in the Table. The active below; the inactive above.

ther, and, if the solution does not become turbid about 90° C. (it should never be turbid below 80° unless large amounts of alkaline salts are present), a few c.c. of dilute acetate should be added. The process may have been clumsily pictured, but it has been practised scores of times, and in experienced hands renders the estimation of nickel in steel the most rapid of all laboratory estimations save colour carbon.

The addition of free acetic acid is not a new idea. "A few drops to prevent the formation of basic acetate of the protoxide" is a familiar injunction. Phillips ("Engineering Chemistry") adds 2 c.c. of 10 E HCl after just dissolving the permanent turbidity. Eggertz (CHEMICAL NEWS, xviii., 232) adds 1½ c.c. HCl. I believe that Jewett gives other and earlier examples in his paper. These additions of HCl serve a good purpose when large amounts of acetate are used, in that they provide more ferric chloride to be decomposed, and leave a smaller excess of acetate to act undesirably on the Mn, Ni, or Co, as the case may be.

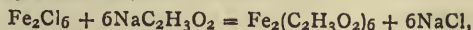
The addition of HCl or HNO₃ to a hydrate-free solution finally resolves itself into the same thing as adding free acetic acid, because, owing to their superior avidity, they react so,—



and a similar change takes place when, through added hydrochloric acid, the proportion of ferric chloride is increased. These theoretical considerations may be verified by noticing that it is indifferent to the turbidity temperature and the accuracy of a separation whether—having a solution containing a known amount of free acid—it be treated by neutralising and adding acetate and acetic acid, or by reversing the order in which the reagents are added, or by mixing them up in any way whatever.

I am persuaded to indulge in the following speculations by the fact that the only author (J. O. Arnold, "Steel Works Analysis," Whittaker and Co., 1895) who makes a special feature of explaining the processes of steel analysis dismisses the acetate separation with "On the addition of ammonium acetate the iron is precipitated as an indefinitely constituted mixture of ferric acetate and hydrate generally known as basic ferric acetate, and some free acetic acid is liberated. The complex reactions bringing about this result are not clearly known." I am encouraged also by the hope that if I miss the mark, which is very possible, I may hit some one else into providing a better explanation.

The precipitation of a solution of ferric chloride may be represented so,—



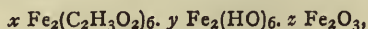
and previous experiment has shown that the proportion of Fe₂Cl₆ and NaC₂H₃O₂ required to effect the precipitation are in fair accord with the equation's requirements.

A solution of ferric acetate on heating is precipitated. The temperature at which precipitation takes place has been shown to be moderately constant. An excess of acetate, however, above that necessary to decompose the ferric chloride effects a separation at a lower temperature, and this decreasing of the temperature takes place in a quite regular manner for increasing acetate.

The precipitated ferric acetate is not a very stable body. In the boiling solution it becomes converted into ferric oxide and free acetic acid. This I surmise is the free acetic acid Prof. Arnold refers to, though I do not know that its liberation is necessarily concurrent with the precipitation, which may take place at very low temperatures. The volume of acetic acid formed is proportional to the

amount of ferric acetate, that is to the amount of ferric chloride previously unchanged into dissolved ferric hydrate by the neutralisation. This explains why, in the copper separations, the solutions free from dissolved ferric hydrate behaved similarly to those having large amounts of acetic acid present. It also provides another reason why the addition of a few c.c. of HCl, as practised by Eggertz, Phillips, &c., lead to a more accurate separation.

Plainly, then, the composition of the "basic acetate" will depend on the proportion of dissolved hydrate—which is dehydrated in the boiling solution—and ferric chloride in the first instance; and then on the extent to which the ferric acetate and hydrate are decomposed by heating. Thus the composition of the precipitate must always be the indefinite—



but the reactions by which it is formed seems to be as understandable as reactions generally are.

With some misgivings I go a step further, and attempt to explain the imperfect separation and the relative positions of the elements in Table XIX. It is remarkable that with the exception of copper, the elements are all members of the iron group; indeed they constitute the whole group.

The explanations required are why an increasing excess of acetate gives a decreasing separation, and why the addition of acetic acid arrests this defection.

Having seen that an addition of soda acetate to a solution of ferric chloride forms ferric acetate, there is no difficulty in understanding that a similar change—and the more readily effected the larger the proportion of alkali acetate—can occur with manganese, zinc, cobalt, &c.

When heated the ferric acetate is precipitated. A similar course is not followed by the other metallic acetates, at least not in pure solutions. Heating at boiling temperatures decomposes ferric acetate in the manner previously mentioned, and through an analogous behaviour of the acetates of the other metals the defective recovery may be accounted for.

I find, on turning to Watts' "Dictionary of Chemistry" (vol. i., p. 9), that "many acetates may be decomposed by water into acetic acid and metallic oxide." This decomposition in the case of aluminic and ferric acetates occurs at 100°, while at 175° the acetates of Mn, Co, Ni, Zn, and Cu are slowly decomposed.

It is true that the acetate separation never involves a temperature beyond 103° to 104° C., and that a defective separation may be obtained at much lower temperatures than that even (60° to 70°), if enough acetate has been used. It is also true that what takes place in a solution of pure salt is frequently modified when the same salt acts in a mixture. I admit the presumption, and yet I beg to be allowed, tentatively at least, to state that these metallic acetates, in company with the iron, are decomposed at a much lower temperature. The evidence, then, for framing a palpable explanation to the first query is complete.

The solubility of one of the products of the decomposition—the metallic oxide—in dilute acids is a sufficient explanation of the action of acetic acid.

If these explanations are true ones we should be able to justify, in an independent manner, viz., by the behaviour of their acetates and oxides, the position of the elements in Table XIX. This I believe we may do fairly satisfactorily.

Perhaps the most effective way will be to let a short Synopsis speak for itself (see p. 223). The data are taken from Watts' "Dictionary" and Comey's "Solubilities." The action of alkali chlorides, which have been occasionally noticed, are also explained.

It is hoped that the foregoing papers may do something to restore alkaline acetates to the favour they deserve.

Attention was drawn months ago to the fact that ammonium acetate was said to be unstable and needed to be

made as required. It may not be amiss to say that the solution then in use is being used now, and so far as I can detect in the working is quite unaltered.

In conclusion, I most warmly tender my thanks to Mr. R. L. Leffler and Mr. Jervis, respectively chemist and co-assistant in this laboratory, for their general interest and appreciated aid in the work.

The Laboratory,
Messrs. Thos. Firth and Sons, Lim.,
Norfolk Works, Sheffield.

ON CARMINIC ACID.

By W. VON MILLER and ROHDE.

CARMINIC acid, dried *in vacuo* at an ordinary temperature, yielded on combustion with lead chromate:—

Carbon	53.65
Hydrogen	4.32

Carminic acid dried in a stream of hydrogen at 115° to 120°:—

Carbon	53.77
Hydrogen	4.27
(Burnt with copper oxide).	

Carminic acid dried *in vacuo* at 80°:—

Carbon I.	53.68
" II.	53.80
" III.	53.75
Hydrogen I.	4.5
" II.	4.7
" III.	4.4

(All burnt with lead chromate).

A sample of crystallised acid prepared according to the procedure of Schunck and Menshutkin gave similar values dried in a current of hydrogen at 115°:—

Carbon	53.69
Hydrogen	4.24

Berichte der Deut. Chem. Gesell., No. 13, p. 1759.

ON THE COMPOSITION OF CERTAIN CANADIAN VIRGIN SOILS.*

By FRANK T. SHUTT, M.A. F.I.C., F.C.S.,
Chemist, Dominion Experimental Farms.

(Concluded from p. 216).

The Maritime Provinces.

THE soils from New Brunswick and Nova Scotia examined by us have been so few in number that it would be unwise to draw from the data conclusions as to the general character of the soils of these provinces. A few examples are here given which, though representative of large areas, must not be considered as the only provincial types; the figures are inserted here to render the data somewhat more complete than they otherwise would be. (See Table VI.).

New Brunswick.

Soil No. 57.—From the Sackville Marsh, at the head of the Bay of Fundy. A clay loam; of interest as an example of a soil area very uniform in character—a fact no doubt due to the origin of the soil, which is practically a tidal deposit. When thoroughly drained, which frees them from salt and improves their texture, these reclaimed Marsh soils are found to be exceedingly fertile. A glance at the

* Read before the British Association (Section B), Toronto Meeting, 1897.

TABLE VI.—Analyses of Soils (Water-free), The Maritime Provinces.

No.	Locality.	Surface or subsoil.	Character of soil.	Potash.	Phosphoric acid.	Nitrogen.	Lime.	Loss on ignition (organic and volatile matter).
57.	Sackville Marsh, N.B.	Surface.	Clay loam	0'16	0'16	0'131	0'13	5'83
58.	Restigouche, N.B.	"	Yellow sandy soil	1'02	0'10	0'113	0'23	5'46
59.	Cumberland, N.S.	"	Sandy loam	0'16	0'09	0'090	0'06	3'37
60.	S.W. Mabou, N.S.	"	"	0'37	0'09	0'212	0'05	6'97
61.	King's Co., P.E.I.	"	"	0'47	0'09	0'106	0'08	5'10

analytical data shows that this is not to be altogether ascribed to large percentages of plant food, and it is more than probable that the fine state of division and the intimate incorporation of the soil particles—due to the manner of the soils formation and deposit—render the elements of fertility more easily obtained and assimilated by the plant.

Soil No. 58.—Balmoral Settlement, Restigouche. A yellow loam, derived principally from the decomposition of felspar, though showing some quartz fragments. The percentage of potash is considerably above that found in average fertile soils—a fact undoubtedly due to the felspathic origin of the soil. With the exception of potash, however, the soil cannot be considered one equal to Canadian soils of average fertility.

Nova Scotia.

Soil No. 59.—A reddish sandy soil from Hansford, Cumberland Co. It is below the average in the more important elements, and to be regarded as a poor soil, but responding well to judicious culture and manuring.

Soil No. 60.—A soil from South-west Mabou, Inverness; very similar in appearance to No. 59, but analysis shows it to be much richer. The small percentage of lime is particularly noticeable in these soils; the knowledge of this fact has assisted towards the economical treatment of them with fertilisers.

Prince Edward Island.

Soil No. 61.—This soil partakes of the same colour as the light red Triassic sandstone from which it is derived, and in this respect at least this sample is representative of the characteristic soil of the province. It differs from the preceding specimens in that it is not a truly virgin soil. Some difficulty was experienced in procuring a sample which had not been cropped or manured; indeed, no guarantee of such could be obtained. This soil, however, is said to fairly represent the unmanured but cultivated soil that extends over a large area in the Eastern portion of the Island. It is light sandy loam, the texture of which is fairly good. Though containing a more than average amount of potash, this soil could not be ranked, from a chemical standpoint, with our richer Canadian soils—possessing but small percentages of nitrogen, phosphoric acid, and lime.

This agricultural province is justly known as a fertile one; and we presume, judging from such data as we have at hand, that this fertility is due rather to good soil texture and favourable climatic influences than to richness of its land in plant food constituents.

The last table (Table VII.) that is presented for your consideration, showing the average amounts of fertilising ingredients in the surface soils that have been examined, taken province by province, has been prepared with no little diffidence. If it were to be interpreted as placing before you data from which deductions could be made as to the average soil fertility of the yet untilled areas of the respective provinces, it might be regarded as misleading. It is not my intention that such a conclusion should be drawn. A hundred or so samples, though they are typical, and, as far as possible, thoroughly representative of large areas, taken from the thousands of square miles of untilled soil in the Dominion, do not afford sufficient basis for such generalisations. They are not provincial averages; they are rather averages from large un-

tilled areas in the several provinces, and may therefore serve to indicate the general character of much of the yet untilled lands in Canada.

TABLE VII.—Analyses of Soils. Averages. Surface Soils.

No. of samples.	Province.	Potash.	Phosphoric acid.	Nitrogen.	Lime.
21	British Columbia	0'42	0'27	0'262	1'17
7	North-West Territories and Manitoba . .	0'44	0'19	0'537	1'08
6	Ontario (Muskoka only)	0'22	0'15	0'135	0'44
6	Quebec	0'44	0'20	0'226	0'52
5	Maritime Provinces	0'44	0'11	0'130	0'11
45	Average of all..	0'39	0'18	0'258	0'66

When we remember that care and judgment were exercised in the selection and collection of these samples, that the analyses were carefully conducted according to modern and approved methods, that very few of the individual samples fall below the standards or limits fixed by agricultural chemists, and that many contained such ample stores of plant food as to warrant them in being classed among the most fertile soils, we may, I think, safely conclude that the data here set forth clearly indicate that while there are many types of soils represented in Canada, there are in all her provinces large tracts of land that, as far as plant food is concerned, compare favourably with the most productive of other countries.

Canada is fast becoming known in the markets of the world as a great food-producing country. Soils rich in plant food and favourable climatic influences are the chief factors that have assisted the Canadian agriculturist in building up this reputation. These are the factors, together with intelligent rational methods of farming and safe cheap means of transportation, that will continue in the future to make agriculture here a prosperous industry. It is therefore gratifying to learn that ample scientific proof is now on record to show that in our virgin soils there is in such abundance the crude materials upon which crops must directly, and farm animals indirectly, thrive.

EARLY AMERICAN CHEMICAL SOCIETIES.*

By Prof. H. CARRINGTON BOLTON.

(Continued from p. 217).

II. The Columbian Chemical Society of Philadelphia.

THE Columbian Chemical Society was founded in the month of August, 1811, by "a number of persons desirous of cultivating chemical science and promoting the state of philosophical inquiry." The names of the gentlemen who attended this meeting are not certainly known, but it may be presumed that they included most of those who were then elected to office; these were as follows:—

* Read before the Washington Chemical Society, April 8, 1897. From the *Journal of the American Chemical Society*, August, 1897.

Patron—Hon. Thomas Jefferson, Esq.

President—Prof. James Cutbush.

Vice-Presidents—George F. Lehman and Franklin Bache.

Secretary—John C. Heberton.

Treasurer—James J. Hamm.

Orator—John R. Barnhill.

And a "Corresponding Committee" of Three: John Barnes, M.D.; John Lynn, M.D.; and Charles Edwards.

Thomas Jefferson's commanding position in the world of science and arts, as well as his literary attainments, well qualified him for the dignified office of patron. He had held the office of president of the most prominent scientific body in the United States (American Philosophical Society) for many years, and only relinquished it to accept the higher one of Chief Magistrate of the Nation. Seventeen months before the founding of the Chemical Society, Jefferson had retired from the presidency, after serving his country eight years, and was living at his country seat, Monticello.

James Cutbush, president of the Columbian Society, was at that time professor of natural philosophy, chemistry, and mineralogy at St. John's College. Little is known of his early history: in 1814 he was appointed to the army with rank of Assistant Apothecary General, and he held the position of chief medical officer of the United States Military Academy at West Point, from June, 1820, to November, 1822; the army being re-organised, he became assistant surgeon and acting professor of chemistry and mineralogy at the same institution, positions which he held until his death, December 15, 1823.

Dr. Cutbush's papers, presented to the Columbian Society, will be considered below. He published also the following:—"On the Formation of Cyanogen in some Chemical Processes not before noted" (*Am. J. Sci.*, vi., 1822), "On the Composition and Properties of the Chinese Fire" (*Ibid.*, vii., 1823), "On the Composition and Properties of Greek Fire" (*Ibid.*, vi., 1822). He was also the author of several books:—"Useful Cabinet" (1808), "Philosophy of Experimental Chemistry" (Philadelphia, 1813), and "A System of Pyrotechny" (Philadelphia, 1825). The last-named is an elaborate work of more than 600 pages, octavo.

George F. Lehman, the first vice-president, published articles in Mitchell's *Medical Repository*, chiefly on medical subjects.

Franklin Bache, the second vice-president, was at that date a youth of only twenty years, who had graduated at the University of Pennsylvania the year before the founding of the society. He was a grandson of Benjamin Franklin, and a member of the distinguished Bache family, which numbered so many eminent men of science. He afterwards became professor of chemistry at the Franklin Institute, and in 1841 at the Jefferson Medical College, which chair he held until his death in 1864. He is remembered also as the author of "A System of Chemistry for the Use of Students of Medicine" (Philadelphia, 1819), and of other chemical treatises.

The constitution adopted by the founders of the society, besides the usual provisions for regulating business, contained some unusual features; the officers included an orator, and Article VII. prescribed:—"An oration on some chemical subject within two months after the commencement of the medical lectures in the University of Pennsylvania, in each year." Since the "Memoirs" published by the society contain no "oration," it is to be feared that the incumbent's efforts were not satisfactory.

Two articles in the constitution deal with fines:—"Every member shall be fined 12½ cents for absence each roll, unless satisfactory reasons be offered." And again, "Any member being elected to office and refusing to serve shall be fined one dollar."

Another notable provision is as follows:—"The society shall appoint, once in each month, some member to read an original chemical essay, for neglect of which the member so appointed shall be fined one dollar." These

finer, with the annual fee of two dollars, were evidently expected to maintain a full treasury.

To become a member of the society special qualifications were prescribed; after being proposed and seconded the candidate "shall read an original essay on some chemical subject, on which any member may speak not more than ten minutes." After this trial of his ability, a two-thirds vote of the members present at a subsequent meeting were required to secure election.

It seems to have been easier to be put out of the society than it was to get in, for "any member behaving in a disorderly manner shall be expelled by consent of two-thirds of the members present."

This mandatory "shall" is used throughout the regulations; the president "shall preserve order," the secretary "shall keep fair minutes," the constitution "shall be revised annually," and so on. To insure against members withdrawing early from a dull meeting, the "secretary shall call the roll at the opening and close of each meeting and mark down absentees," each of whom is then fined 12½ cents as stated. Never did a society undertake to control its members with more stringent rules!

The members who subscribed to these regulations were divided into two classes, "Junior" and "Honorary" members, the former corresponding to a class which would now be styled "Associates," and the latter including both American and foreign chemists of distinction. The junior members numbered thirteen, the honorary members numbered sixty-nine, thirty-one of whom were Europeans. The home list included most of those chemists, then living in America, whose labours contributed largely to the foundations of the science in the New World. Brief notices of some of the members will serve to summarise the status of chemistry in the United States for the years 1811 to 1813.

Dr. Benjamin Smith Barton (1766-1815) held the chair of medicine, natural history, and botany in the University of Pennsylvania. Dr. Barton has been called by his admirers "the father of American natural history," though there are other claimants for this honourable designation—Mitchell, of New York, and Thomas Jefferson. Dr. Browne Goode, writing of Barton, says he, of all the early Philadelphia naturalists, "had the most salutary influence on the progress of science." He was a leader in the American Philosophical Society, and an agreeable writer on natural history topics, and, though he made no contributions to chemistry, was a worthy member of the society.

Dr. Archibald Bruce (1777-1818), one of the pioneers of mineralogical science in America: he had established the *American Mineralogical Journal* one year before the date of which I write. His analyses of minerals mark him as a skilful chemist. He held the chair of mineralogy in Columbia College, New York.

Joseph Cloud (1770-1845) was assay master of the United States Mint in Philadelphia, and already distinguished by his researches on palladium (1807).

Thomas Cooper (1759-1840), born in London, had come to America in 1792, with his friend Priestley, whose radical views in politics and religion he shared. Dr. Cooper wrote much on political, ethical, and philosophical subjects, and published some essays on chemistry. In 1811-14, the period of the Columbian Chemical Society, he held the chair of chemistry at Dickinson College, Carlisle, Pa., and in 1819-34 he held the same position at the College in Columbia, S.C., of which he afterwards became president.

Dr. John Redman Coxe was professor in the medical department of the University of Pennsylvania, having succeeded Dr. Woodhouse. He made several original observations in chemistry published in current periodicals.

Dr. Edward Cutbush (1772-1843) was surgeon of the United States Navy and professor of chemistry in the medical school of the Columbian University, Washington (1825-27). He has another honourable claim to distinction, having been the founder in 1819 of the

Columbian Institute for the Promotion of Arts and Sciences in Washington, a sort of precursor of the Smithsonian Institution.

Passing with brief mention Dr. Elisha de Butts, professor of chemistry in the College of Maryland, Prof. Benjamin de Witt, of New York, and Dr. John Syng Dorsey, already mentioned as a member of the society founded in 1792, we reach the more familiar name of Dr. John Griscom, "the acknowledged head of all teachers of chemistry in New York City," for more than thirty years.

The next name in the alphabetical list of members is that of Robert Hare, professor of natural philosophy in the University of Pennsylvania, whose career we have already noticed.

Dr. David Hosack (1769-1835), professor of botany and materia medica in Columbia College, New York, is best known as the founder of the first public botanic garden in the United States, in 1801. His contributions to science were chiefly in medicine. The tragic circumstances of his death have been nearly forgotten; he died of shock at the disastrous conflagration in New York City in 1835, which swept away his property to the value of \$300,000.

Dr. Henry Jackson, professor of chemistry at Athens College, Georgia, is followed by His Excellency James Madison, LL.D., President of the United States of America, whose name added lustre to the rolls of the society, but whose claim to the membership can only be based on extensive general information.

Dr. John Manners, of Philadelphia, affixes to his name the initials F.A.N.S., the Academy of Natural Sciences, having been founded one year before the printing of the list of members. His contributions to the Chemical Society will be noted below.

Dr. John Maclean (1771-1840) was the first professor of chemistry in the College of New Jersey, now Princeton University, to which chair he was elected in 1797. In accordance with the prevailing custom, he also gave the instruction in astronomy, mathematics, natural philosophy, and natural history; this fact is ample apology for his not appearing in the ranks of original investigators. Prof. Maclean published in 1797 "Two Lectures on Combustion," in which he upheld the views of Lavoisier, as opposed to the phlogistic theory maintained by Dr. Priestley.

The Hon. Samuel L. Mitchell, M.D., F.R.S.E. (1764-1831), professor of chemistry and natural history in Columbia College from 1792, was active in many branches of scientific research. In 1798 he established the *New York Medical Repository*, which for sixteen years was an influential organ in recording and diffusing progress in general science, as well as in medicine. His zeal for science did not prevent his taking part in national affairs, for he occupied a seat in the senate of the United States from 1804.

Dr. Thomas D. Mitchell, F.A.N.S., was one of the most active members of the society, frequently contributing to its memoirs.

Passing by Dr. John C. Osborne, professor of the institutes and practice of medicine in Columbia College, New York; Dr. Joseph Parish, of Philadelphia; Mr. Robert Patterson (1743-1824), professor of mathematics and lecturer on natural philosophy in the University of Pennsylvania, director of the United States Mint, and afterwards (1819) president of the American Philosophical Society; Dr. Nathaniel Potter, professor of the theory and practice of medicine, University of Maryland, we reach the eminent Dr. Benjamin Rush, professor of the institutes and practice of medicine in the University of Pennsylvania. Dr. Rush (1745-1813) has been characterised by Benjamin Silliman as "undoubtedly the first Professor of Chemistry in America, his appointment dating August 1, 1769. In his busy life, besides his professorial chair, he filled the positions of surgeon-general of the United States Army (1777), treasurer of the Mint,

president of the Society for the Abolition of Slavery, vice-president of the Bible Society of Philadelphia, and conducted a large medical practice in the same city.

(To be continued).

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, October 29th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

PROF. STROUD exhibited and described the "*Barr and Stroud Range-finder*." The problem of finding the distance of a given object at sea, or in the field, is complicated by the shortness of the trigonometrical "base" and by restrictions of time. As a rule the apparatus must be self-contained, and "snap-shot" readings are obligatory, —i.e., the range has to be determined from a single instrument and from a single observation. At 3000 yards the errors must not exceed 3 per cent. In foggy weather, or when viewing a nebulous object, this degree of precision is difficult to attain; but under favourable circumstances the authors have determined ranges, at that distance, within 1 per cent of accuracy. At shorter ranges measurement is more exact; thus an object at about 2000 yards may be estimated to within about 12 yards. Prof. Stroud gave some account of the history and of the general methods employed in these instruments. Two images of the distant object, preferably of a line such as a flag-staff, are received respectively upon two mirrors, two lenses, or two prisms, placed one at each end of a fixed support. From each of these the light is then directed towards the middle of the instrument, where the two images, after further reflection, are viewed by one eyepiece. The optical system has finally to be adjusted so that the two images, as now seen in the eyepiece, lie in the same straight line. In the instrument designed by the authors this coincidence is attained by translating a small prism parallel to the axis of the supporting rod. The extent of this translation is a measure of the range. Both eyes are used; the right for bringing the two images into alignment; the left for "finding" the object through a small field-glass, and for reading the scale of distances. At night, sightings have to be taken from "points" of light, and, as these are unsuited to measurement, the authors convert them into "lines" by the use of cylindrical lenses. Various devices are introduced to prevent over-lapping of the images. The instrument is about 5 feet long and tubular in form; it is made of copper, so as to have high thermal conductivity to reduce differential heating. Within the outer tube is the interior supporting rod, designed to equalise so far as possible the effects of interior radiations. Several forms of "separating" prisms were exhibited: the best for the purpose consists of two "reflecting" prisms; these receive the two rays, and direct both of them into a third prism, whose angle lies in the space between the angles of the others.

MR. BARR drew attention to the gimbal arrangement and the three struts that keep the supporting rod central in the tube. To give some idea of the precision and scope of the range-finder, he observed that they were there using the equivalent of a 25-ft. "circle," and their measurements were comparable to the measurement of 20 secs. of angle, on such a circle. The instrument is handled by ordinary seamen, and stands rough usage on board ship for years without injury.

PROF. STROUD then exhibited a "*Focometer and Spherometer*." He explained that in determining curvatures and focal lengths some telemetric method was necessary, and that, owing to want of parallelism of the beam and

duplication of images, a short-focus telescope was always an inefficient telemeter. For the measurement of inaccessible lengths it was therefore better to use some simple form of "range-finder." Such an apparatus could be made with a set of small mirrors, arranged in such a manner as to direct two images of the distant object into an eyepiece, with a fixed prism in the path of one of the incident beams. By sliding this instrument along the optical bench, one position could always be found at which the two images, as seen through the eyepiece, were in coincidence. He also described a method for determining curvature by interposing a plate of plane glass between the curved mirror and a source of light.

Mr. ACKERMANN exhibited two experiments: (1) The blowing-out of a candle-flame by the air from a deflating soap-bubble. The bubble was blown at the mouth of an inverted beaker by breathing into a hole cut out at the top. This hole was then presented to the flame, and the flame was immediately quenched. But if the bubble was blown from ordinary air, with bellows, the flame was merely deflected without being extinguished. (2) It was shown that a miniature boat, provided with a false stern, consisting of a linen diaphragm, could be propelled by filling the hollow stern-space with ether, or with some liquid similarly miscible with water. The motion is due to the continuous release of surface-tension behind the boat.

Prof. Boys said that when he tried, some years ago, to blow out a candle with a soap-bubble filled with common air, he found the operation very difficult,—so difficult that, having once succeeded, he never repeated the attempt. It had not occurred to him, as it had to Mr. Ackermann, that the CO_2 present in the breath played a part in the quenching. With regard to the second experiment, he had seen a small boat propelled by dissolving camphor astringer, but he thought the use of a liquid for that purpose was a novelty.

The PRESIDENT proposed votes of thanks, and the meeting was adjourned until November 12th.

NOTICES OF BOOKS.

The Principles of Chemistry. By D. MENDELEEFF. Translated from the Russian (Sixth Edition) by GEORGE KAMENSKY, A.R.S.M., of the Imperial Mint, St. Petersburg; Member of the Russian Physico-Chemical Society. Edited by T. A. LAWSON, B.Sc., Ph.D. In Two Volumes. London, New York, and Bombay: Longmans, Green, and Co. 1897.

It is not surprising that a new edition of D. Mendeleeff's *opus magnum* has been required and has accordingly appeared.

The introduction, besides other useful and necessary matter, contains correct definitions of a variety of terms occurring in technical and scientific discussions, and at present grossly misused. A signal instance may be found in the word "phenomenon" and its paronyms.

Of the principles or generalisations of chemical science, the Periodic Law is justifiably spoken of as the most important step in the development of chemistry since the establishment of the atomic theory. The author does not discuss the various, and to some degree conflicting, claims of different chemists to the honour of the first or original discovery.

In the introduction it may be questioned whether the phlogistic theory and its leaders Becher and Stahl are not treated with a too benevolent neutrality. Indeed, Professor Mendeleeff is evidently no controversialist; he draws a parallel between Lavoisier and Dalton on the one hand, and Copernicus and Kepler on the other; but he recognises that the molecular world has not yet found

its Newton. He calls attention to the study of Professor Kononoff on compact phenomena.

Concerning the waters of well known rivers, the author points out that the Neva is remarkable for the small amount of solid matter which it contains, *i.e.*, per cubic metre 55 grms., of which 32 grms. are incombustible and 23 grms. organic.

The constitution of salts was a matter of much discussion. The binary theory dates from Rouelle and Lavoisier; the electro-chemical phase of the question was especially upheld by Berzelius; and the hydrogen theory, which is at present dominant, is due to Davy and Liebig.

Ozone has also proved a bone of contention; the question of its presence in atmospheric air is still open. Ilosvay de Ilosva concludes, from a prolonged course of experiments, that the phenomena recently ascribed to ozone are probably due to nitrous acid.

In Volume II., after a table of the periodicity of the elements, we find an account of the elements as demonstrating the author's theory. We find the relations of platinum, palladium, and nickel, and again gold, silver, and copper, distinctly brought into view.

The author contends that the periodic law enables us to see a regularity in the variation of all chemical and physical properties of elements and compounds, and has rendered it possible to foretell the properties of elements and compounds as yet uninvestigated by experimental means.

Copper, silver, and gold melt far more easily than platinum, palladium, and nickel, whilst zinc, cadmium, and mercury melt still more easily. Nickel, palladium, and platinum are very slightly volatile; copper, silver, and gold more volatile; whilst zinc, cadmium, and mercury are among the most volatile metals. Zinc is oxidised more readily than copper, and is reduced with more difficulty, and the same holds good for mercury as compared with gold, whilst the properties of cadmium are intermediate in their respective groups.

Attention is briefly called to the want of two elements whose existence is not sufficiently demonstrated—ekacadmium and actinium.

Crystalline boron is very closely analogous to diamond, *i.e.*, crystalline carbon. It has the lustre, the high refractive power of the diamond, with which mineral it also competes in hardness.

We find here the comparative analysis of four soils, among which the famous "black earth" claims the first rank on account of its richness in potassium, phosphoric acid, and nitrogen.

As regards the localities for bauxite, the north-west of Ireland is unfortunately omitted. Graham's characteristic names for the different states of aluminium hydroxide—"hydrogel" and "hydrosol"—are quoted with approval; and the part played by aluminium acetate in the dyer's procedures is explained.

The practical importance of the aluminium alloys, especially that with copper, is duly appreciated. Pure aluminium is now employed only for objects which require hardness in combination with a low specific gravity. Notes are given on the separation of the rare metals as indicated by Sir William Crookes and others. Doubts are entertained as to whether Welsbach's fractionation of didymium into neodmium and praseodymium can be regarded as final. Becquerel resolves didymium into six individual elements and Sir William Crookes has proceeded still further.

The reader is reminded that the researches of Graham point to the transition from inorganic to organic compounds.

The author gives the general average distribution of phosphoric acid in soils and earthy substances in nature as 1 to 10 parts in 10,000 parts. Its presence in excess is no less pernicious to vegetation than its absence. The red variety—commonly, but erroneously, known as amorphous phosphorus—does not appear to be poisonous. There is a further variety known as metallic phosphorus;

t stands nearer to nitrogen than the yellow variety. The spontaneous inflammability of the hydride PH_2 is mentioned as an interesting fact; possibly the formation of a trace of this variety may account for the phenomena of the *Ignis fatuus*.

The difference between the reactions of ortho-, meta-, and pyro-phosphoric acids the author considers of the deepest interest as regards the theory of hydrates and solutions, which has not yet been fathomed.

Though arsenic is closely analogous to phosphorus, it has a certain resemblance and even isomorphism with the corresponding compounds of sulphur.

The arsenical mirror is a special variety of metallic arsenic, whilst the brown product found simultaneously in the Marsh apparatus is AsH .

The whole of this work has the merit of presenting the loftiest generalisation in harmony with, and based upon, a careful study of facts.

Elements of Chemistry. By RUFUS P. WILLIAMS. Pp. 412. Boston, U.S.A., and London: Ginn and Co., The Athenæum Press. 1897.

THE method of teaching adopted in this book is the result of the author's experience with some 2500 pupils; one great point being his endeavour to make the work have a real fascination for the student by awakening in him his sense of enquiry.

What proportion of time should be devoted to laboratory work has long been a matter of discussion. Students differ considerably, but after some experiments on a large scale, Mr. Williams concludes that, with four or five hours a week for chemistry, less than half should be spent in laboratory work.

The book is divided into forty-one chapters, so we cannot take them seriatim; suffice it to say that the whole subject of elementary chemistry is carefully explained and amply illustrated by formulae, equations, and exercises.

The subject of valence, Chapter X., may be noted as being treated in a specially clear manner.

In Chapter XLI. a brief reference is made to microbes and bacteriology, but the subject is naturally too far advanced to be thoroughly treated in a book for beginners.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Moniteur Scientifique,
Series 4, Vol. xi., September, 1897.

Application of Electrolysis to the Manufacture of Inorganic Products.—L. GOURWITSCH.—The applications of electrolysis to inorganic chemistry are not very varied; they consist for the most part in decomposing salts, and the secondary reactions which occur are only very rarely synthetical. The object of this paper is to review the present state of the question, and numerous references are made to the original papers. The principal subjects touched upon are the electrolytic manufacture of alkalis and chlorine, by the electrolysis of melted chlorides, by methods founded on the use of mercury as the cathode, and by the electrolysis of hydrochloric acid. The electrolytic manufacture of chlorates, hypochlorites, persulphates, permanganate of potash, chromates, bichromates, &c., is also described.

Chemical Modifications which take place in Fruits during their Growth.—C. GERBER.—The author finds that the acids in fruits disappear, giving off at the same time more carbonic acid gas than they can borrow from the

atmospheric oxygen; they form hydrates of carbon. The tannins disappear, on the contrary, when the respiratory quotient is below unity. They do not form hydrates of carbon: so long as tannins exist the fruit will not soften. As soon as the tannins have disappeared softening commences; then follows obstruction of the intercellular meatus, alcoholic fermentation, and the formation of perfumed ethers; at the same time the respiratory quotient becomes greater than unity.

Estimation of Sulphuric Acid. Gravimetric and Volumetric Methods.—F. MARBOUTIN.—Will be inserted at length.

On some New Sulphurised Colouring Matters.—R. VIDAL.—In 1893 the author patented a process for the manufacture of black dyes for cotton, resulting from the action of sulphur on hydroquinone in the presence of ammonia. Some time after that the Société des Matières Colorantes, at St. Denis, took out a patent for the same purpose, by the action of sulphur on paramidophenol. This reaction the author claims to depend entirely on his first patent. It is a very general reaction, and can be applied to all the di- and tri-substituted derivatives of benzene and naphthalene which have two amidised functions, or one amidised and one hydroxylised function, situated in para.

MISCELLANEOUS.

The Commercial Development Corporation, Ltd.—This Company has been formed primarily to purchase, and thereafter work and commercially develop, Mr. J. G. A. Rhodin's patent improved Electrolyser, which, the Prospectus states, is "an improved process for the electrolytic production of alkali and bleaching-powder, which are two of the staple commodities of the world in constant and ever-increasing demand." Patents have already been granted or applied for in Great Britain, the Colonies, the United States, and the various Continental countries. The purchase price for the patents has been fixed at £90,000, payable as to £20,000 in fully-paid shares, £10,000 in deferred shares, and £60,000 in cash. Dr. John Hopkinson, F.R.S., has been retained as Technical Adviser. The Company also proposes to acquire, as occasion arises, other patents, business, and properties. The capital is fixed at £200,000, divided into 190,000 Ordinary Shares of £1 each and 10,000 Deferred Shares of £1 each. The present issue consists of 90,000 Ordinary Shares of £1 each and 10,000 Deferred Shares of £1 each, of which 70,000 Ordinary Shares are offered for subscription; £100,000 being reserved for further issue if and when required. The Prospectus states that "sufficient capital for the present needs of the Company having been subscribed by the Directors and their friends, the Company will at once proceed to allotment on the closing of the lists," the date of which is fixed at Monday next, November 8th, at 4 p.m., for both Town and Country.

Brussels International Exhibition.—The Horsfall Furnace Syndicate have been awarded, at the Brussels International Exhibition, a Gold Medal for their patent Refuse Destructors, and a Bronze Medal for their patent Smoke-consuming Boiler Furnace.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 1st inst., Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, presiding. The following was elected a Member:—Mr. John W. Woodall, J.P. The special thanks of the Members were returned to Dr. A. J. Hipkins for his valuable present of the Collection of Tuning-Forks made by the late Dr. Alexander J. Ellis, F.R.S., M.C.I.

Technical Instruction at Manchester.—In July and August of the present year a Committee of gentlemen, nominated by the Technical Instruction Committee of the city of Manchester, was deputed to visit certain institutions and schools on the Continent devoted mainly to scientific and artistic instruction as applied to commercial and industrial pursuits. Nine towns in Germany and Austria were visited, and the report of the Committee is now issued. The Act of 1889 establishing technical instruction in England and Wales has awakened much interest throughout the country on the subject of industrial scientific teaching and training; it has also directed the attention of the educational authorities of foreign countries to the efforts being made in England, with the consequence that there has been a considerable development, throughout Germany at least, of educational means and resources. To this may be attributed Germany's great commercial and industrial progress of recent years. It was therefore considered to be of the highest importance to see what the Continental countries have done of late; hence the appointment of this committee. That Germany is in a prosperous condition, due to her successful manufacturing and commercial enterprise, is evident in the extension of her cities, the making of new streets, and the erection of handsome buildings which is going on in every town, and it seems certain that it is the technical teaching which is at the root of this surprising development. We sincerely hope that the report of this Committee will arouse many of our large towns from the apathy which they now show, and be the means of further endeavours for the maintenance of our commercial supremacy, once unchallenged, but now so seriously threatened, both by competitors abroad and so-called "labour leaders" and Trades Unions at home.

NOTES AND QUERIES.

Enamelling Cast-Iron Pots.—I should be obliged for information as to the best material and process for enamelling cast-iron pots to withstand 180° Tw., or the names of enamel pot makers.—ENAMEL.

MEETINGS FOR THE WEEK.

FRIDAY, 12th.—Physical, 5. "On the Isothermals of Ether," by J. Rose Innes. "On the Variation with Temperature of the Electromotive Force of the H-form of Clark Cells," by F. S. Spiers and F. Twyman.

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LENT TERM—Monday nearest to the 15th of January to the second Saturday in April.

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CIVIL SERVICE COMMISSION.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1981.

THE SPECTROGRAPHIC ANALYSIS OF MINERALS AND METEORITES.*

By Professor W. N. HARTLEY, F.R.S., and HUGH RAMAGE.

In the course of a spectrographic study of the basic Bessemer flame at Middlesborough the element gallium was found in the iron and was traced to the Cleveland clay ironstone. The process employed in the analysis of the ore was partly chemical and partly spectrographic, and other samples of ores were examined in a similar way (*Proc. Roy. Soc.*, lx., p. 393). The results were satisfactory as far as the detection of gallium was concerned, but the process occupied too much time. The same ores were then examined by a simpler process, in which 0.5 gm. of the powders were rolled in filter-paper and heated in the oxyhydrogen flame, the spectrum of the flame being meanwhile photographed. This operation lasted only about three minutes, and the test for gallium was almost as sensitive as the combined chemical and spectrographic method performed on a much larger quantity, and, in addition, it revealed the presence of many other elements.

A large number of minerals, commercial products, meteoric irons, and meteorites have been examined by this process and very interesting results have been obtained (*Journ. Chem. Soc.*, 1897, pp. 533 and 547). The spectrograph used in this work was described by the aid of a photograph, and spectrograms taken with the instrument were projected directly on the screen.

The simple character of the oxyhydrogen flame spectra as compared with spark spectra was illustrated by examples of the spectra of iron. The former spectra are such that, with the aid of a superimposed spark spectrum of two alloys which give easily recognised lines, the elements may be identified without scale or micrometer measurement. In a mineral or complex mixture of substances the presence or absence of about one-third of the elements may in this way be decided upon in a few moments.

Spectrograms of the following elements were projected on the screen and the lines by which they are most easily recognised indicated:—

Lithium, sodium, potassium, rubidium, and caesium.
Copper, silver, and gold.
Calcium, strontium, and barium.
Zinc and cadmium.
Gallium.
Manganese.
Iron, cobalt, and nickel.

The spectrograms of a number of minerals and meteoric irons were also exhibited, special attention being drawn to the lines of the more interesting constituents.

Tabulated results of the analyses of 169 minerals were given, as in *Engineering*, lxiv., p. 395, and of 13 meteorites, as in a paper read before the Royal Dublin Society in May last, but not yet published in the *Proceedings*.

The Sanitary Institute.—A Sessional Meeting of the Institute will be held at the Parkes Museum, Margaret St., W., on Wednesday, November 17th, at 8 p.m., when a Discussion will take place on "The Pollution of Water Supplies by Encampments of Hop-Pickers, Casual Workers, Tramps, &c." To be opened by Prof. W. H. Corfield, M.A., M.D. (Oxon), F.R.C.P., in reference to the Dangers of Pollution of Municipal Water Supplies; and by Miss M. A. Chreiman, in reference to the Sanitary Control of Hop-Pickers, &c. The chair will be taken by Sir Douglas Galton, K.C.B., D.C.L., LL.D., F.R.S.

* Abstract of a Paper read before the British Association (Section B), Toronto Meeting, 1897.

THE INTERACTION OF HYDROGEN SULPHIDE AND COPPER SALTS.

By JOHN B. COPPOCK, F.C.S.

In the *CHEMICAL NEWS* (vol. lxxiii., No. 1906) I gave an account of some results obtained in connection with the interaction of hydrogen sulphide and copper sulphate,—investigations which took their origin in the statement that cupric sulphide (CuS) was not produced in the interaction, but Cu_4S_3 .

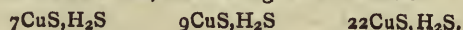
Dr. Brauner in an article (vol. lxxiv., p. 99) pointed out that the precipitates he obtained in working upon this subject contained varying proportions of copper and sulphur, that their composition was not always Cu_4S_3 , and they never contained copper and sulphur in the atomic proportion CuS , except when the original precipitate was left unwashed with carbon bisulphide. Then the precipitate contained copper and sulphur "in the exact atomic proportions $\text{Cu} : \text{S} = 1 : 1$," but some of the sulphur existed in the free state.

Thus it appeared as a point worth noticing that a substance in the process of formation should exert an influence in forming a mechanical mixture of itself and the uncombined negative radicle in exact atomic proportions. This may be but a coincidence, but it suggests the idea that when a lower sulphide is being produced, if free sulphur is formed at the same time, then the lower sulphide and the free sulphur are in the exact atomic proportions required by the higher sulphide.

Analyses of these unwashed precipitates led me to the conclusion that the free sulphur mixed with the copper sulphide precipitate was a very variable quantity, which depended very much upon the time the current of gas was passing through the copper salt solution.

There can be no doubt that the precipitate yielded by the interaction of copper salts and hydrogen sulphide do not always agree with the formula CuS .

Dr. Brauner points out that these precipitates can be represented dualistically as a mixture of Cu_2S and CuS . Several of the precipitates obtained by me, deviating from the formula CuS , I found, after reading Linder and Picton's work (*Chemical Society's Transactions*, 1892), could be represented as compounds of their suggested type. They showed that compounds of the form $n\text{CuS}, \text{H}_2\text{S}$ were formed by interaction of the two above specified substances, establishing the existence of—



What interpretation is to be put upon the fact that the precipitate yielded by the interaction of hydrogen sulphide and copper salts—omitting free sulphur from consideration, as undoubtedly this may be removed by washing—does not always agree with the formula CuS .

Dr. Brauner argues in favour of the production of mixtures of Cu_2S and CuS , which he says finds support, in the action of hydrogen sulphide upon solutions of antimonious and arsenic salts, where mixtures of the lower and higher sulphides are formed, and the tendency which copper has of forming sulpho-salts, as is the case with arsenic and antimony.

That copper does form sulpho-salts—double sulphides—is a fact, but whether it forms a basis for any analogy with arsenic and antimony is doubtful. From considerations of relationship one would expect the production of the compounds $n\text{CuS}, \text{H}_2\text{S}$ to be most likely, as demonstrated by Linder and Picton's work. If hydrogen finds its proper place at head of Group I. (Periodic Law), we should expect it to resemble the other high members of this group, and give combinations with cupric sulphide, as in the cases of sodium and potassium sulphides. Double sulphides are thus known, but they are not formed with the ease and direct manner as are the double sulphides of antimony and arsenic, whose sulphides are very soluble in alkali sulphides, but cupric sulphide is relatively insoluble. The very little analogy between the

two cases does not warrant the idea of an exact resemblance in the interaction of antimonious and arsenic salts with hydrogen sulphide and copper salts with hydrogen sulphide. Moreover, the sulphides of nickel and mercury show a slight solubility in alkali sulphides like copper.

The following method gave almost uniform results in testing whether cupric sulphide could be prepared by action of hydrogen sulphide upon copper salt solutions:—

An unweighed quantity of copper sulphate was dissolved in water, acidified with nitric acid. Nitric acid was added on the idea that any reducing action hydrogen sulphide may possess would be spent upon the acid rather than upon the copper sulphate, and so preventing the production of cuprous sulphide.

The copper sulphate solution was run gradually into hydrogen sulphide solution. The precipitate, after collection, was washed in a cylinder by agitation with dilute acid, then washed free from acid, then left in contact with carbon bisulphide for days, seven at least, during which shaking up went on at times; finally it was washed with alcohol. The precipitate was of a very dark olive-green colour. It is needless to say during these operations oxidation was reduced to a minimum, and apparently none went on.

The precipitate was then dried in an atmosphere of carbon dioxide at a temperature never higher than 100°, inasmuch as it has been shown cupric sulphide reduces to cuprous sulphide when heated to 130°. The precipitate was then fractionated upon the idea that if there were Cu_2S or free sulphur in it such might not be uniformly diffused. Weighed portions were taken and determined by conversion into sulphate and precipitation as barium sulphate, and other portions were digested with strong hydrochloric added, got into solution, oxidised, and the copper determined as cupric oxide. In the oxidation to barium sulphate a little sodium chloride was added, to convert any free sulphuric acid into sodium sulphate, inasmuch as free sulphur was liberated. The following results, and others, were got:—

CuS taken.	CuO found.	Theory for CuS.
0.33	0.2741	0.2744
0.56	0.4652	0.4654
CuS taken.	BaSO_4 found.	Theory for CuS.
0.276	0.6775	0.6779
0.432	1.0616	1.0596

We may therefore conclude that cupric sulphide may be prepared by this interaction. Linder and Picton, in 1892, concluded that in the presence of acid the molecule $(\text{CuS})_n$ was formed as a final breaking-up product of compound between it and hydrogen sulphide.

These investigations have suggested the possible power that precipitates, particularly flocculent ones, may have of retaining finely divided sulphur and hydrogen sulphide, physically or chemically, in their pores, and experiments are contemplated under these headings with other substances than sulphides.

Chemical Laboratory,
Harris Institute, Preston.

ON THE ESTIMATION OF SULPHURIC ACID.

GRAVIMETRIC AND VOLUMETRIC METHODS.*

By FELIX MARBOUTIN.

THE large number of volumetric methods for the estimation of sulphuric acid contrasts strangely with the one gravimetric method used. We propose to give a short résumé of the more frequently used volumetric methods, classifying the various processes; we will then describe

in detail a volumetric method which has given us excellent results in the analysis of water. We also think it advisable to describe in some detail the gravimetric method we employed, as it has served as a check on our volumetric method.

1. *Gravimetric Analysis.*—A volume of water, sufficient to contain 50 to 100 m.grms. of sulphuric anhydride, is concentrated to about 100 c.c. after the addition of a few drops of hydrochloric acid. It is kept at a temperature near boiling, without, however, quite reaching that point; we then add, drop by drop, 10 c.c. of a solution of chloride of barium, of 175 grms. of the crystalline salt per litre. This done, we leave it in a warm place (40°) for twelve hours, wash by decantation with warm water, until nitrate of silver no longer gives a precipitate in the washings; throw on a filter, and again wash with warm water, then place the funnel in an oven, and leave it at 110° for three hours; then put the filter and its contents in a platinum crucible, the point of the filter upwards, and calcine strongly in an oxidising flame; there is then no risk of reducing the sulphate of barium.

This method gives perfect satisfaction; the precipitate is granular, and never passes through the filter, and the use of sulphuric or nitric acid to re-convert the sulphide of barium to sulphate is rendered unnecessary, an appreciable advantage when there are many estimations to make.

In certain cases (highly coloured waters) it is as well to make sure that the precipitate does not contain any foreign matters; for this purpose, after weighing, we fuse with carbonate of soda, then dissolve in water. The solution, acidulated with hydrochloric acid, is precipitated as before.

Ferrous or ferric salts might give a precipitate containing iron, by the formation of a hydrated ferrico-barytic sulphate; it is therefore advisable to get rid of these salts if they are present in too great quantity.

When concentrating large volumes of water it is as well to use a vacuum, for the sulphurous products from the gas flame may easily cause the formation and absorption of 10 to 14 m.grms. of sulphuric acid in six hours if the vessel is uncovered. In the case of sewage waters, &c., it is necessary to get rid of the organic matters by evaporating with fuming nitric acid, or by treating with bromine; but care should be taken not to use an excess or to carry the evaporation too far.

2. *Volumetric Analysis.*—Many methods have been proposed; some specially for alkaline sulphates, others for cases when no chlorides are present, others again are almost universal, but all require several titrated solutions, which causes a considerable amount of uncertainty. Our method, which we shall describe after reviewing those more generally known, presents, we think, certain advantages over those in common use.

I. Direct Method by Chloride of Barium.

A. Houzeau in France, and Wildenstein in Germany, proposed precipitating the sulphuric acid by a titrated solution of chloride of barium, the end of the operation being determined by the absence of precipitation. The determination point, however, is very difficult to decide, and it must not be forgotten that there is a neutral point, where—though there is still some sulphuric acid present—no precipitate is formed unless excess of chloride of barium be added.

II. Direct Alkalimetric Methods.

1. *Using Salts of Barium.*—F. Mohr added an excess of carbonate of barium in the presence of a current of carbonic acid; the alkaline sulphates form sulphate of barium, and the alkaline carbonates formed are estimated by means of a titrated acid solution. Böhlig has improved this method by working at the boiling-point, but it is not applicable except to the sulphates of the alkaline earths.

2. *Using Salts of Strontium.*—Rose showed the com-

* Abridged from the *Moniteur Scientifique*, Series 4, vol. xi, September, 1897.

plete transformation of sulphate of strontium into carbonate, by digesting with alkaline carbonates. Mohr and Classen devised a method founded on this principle. The sample was acidulated with HCl, treated with chloride of strontium, and an equal volume of alcohol added. The washed precipitate is digested with carbonate of soda, and dissolved in excess of titrated HCl. The excess is measured with a titrated potash solution.

3. *Using Salts of Lead.*—The sulphate of lead obtained by precipitating with the nitrate is washed, transformed into carbonate by digesting with carbonate of ammonium, dissolved in excess of titrated nitric acid, and the excess of acid measured with titrated soda. This method cannot be used in the presence of chlorides.

4. *Using Caustic Baryta.*—J. Grossmann precipitates the sulphuric acid by caustic baryta; the excess of hydrate of baryta is treated with a current of carbonic acid, the excess of which is driven off by boiling. The soda is titrated alkalimetrically.

5. *Transforming the Precipitated Sulphate into Sulphide.*—According to Linossier the acid may be separated from the solution as an insoluble compound and converted into sulphide. This latter is titrated alkalimetrically with a normal soda solution, using Poirrier orange as indicator. In the case of sulphuric acid it is precipitated by acetate of lead, and changed into sulphide with sulphuretted hydrogen.

III. Indirect Alkalimetric Methods.

1. C. Mohr precipitates the sulphuric acid by an excess of chloride of barium, in a neutral or very faintly acid solution. The excess of chloride of barium is treated with carbonate of ammonia or soda, the precipitate is washed thoroughly, and the carbonate, dissolved in an excess of titrated acid, is determined by means of a titrated alkaline solution. Ad. Clemm has made this method more practicable by adding, after precipitating with chloride of barium, a quantity of carbonate of soda capable of precipitating all the chloride of barium used. The excess of carbonate of soda remaining measures the quantity of sulphuric acid present.

2. Böhlig precipitates with carbonate of baryta in presence of a current of carbonic acid; the alkaline sulphates are transformed into bicarbonates, and the sulphuric acid is precipitated. The carbonates obtained by boiling are estimated with titrated acid in excess, and titrating back with an alkaline solution.

3. Knöfler makes the sample neutral with titrated hydrochloric acid, then boils to drive off the carbonic acid, and makes the solution neutral to phenolphthalein by means of a titrated solution of carbonate of soda. The sulphuric acid is precipitated with titrated chloride of barium, the phthalein showing when the precipitation is complete; he then adds a slight excess of chloride of barium, which is estimated. Altogether this method requires four titrations, each liable to error, and we do not think it can be employed advantageously in most cases. It is worth noting that readings with phenolphthalein in the presence of carbonates should be made in boiling solutions.

IV. Methods by Precipitation with Chloride of Barium, and Measurement of the Excess.

1. Wildenstein proposed to neutralise the solution from which the sulphate had been precipitated, and to measure the excess of chloride of barium by means of chromate of potassium, the end of the operation being shown by the yellow colour of the liquid; the results, however, are never exact.

2. Precht, using an acid solution, transforms the chromate into bichromate, which he treats with soda to change the colour from red to yellow; the excess of chromate is then measured with a salt of iron, by the touch test. This latter operation allows of the use of cloudy solutions.

3. A. Pellet precipitates the sulphuric acid with an

excess of chloride of barium; the excess is then precipitated by a quantity of potassic chromate equivalent to the total quantity of chloride of barium used. The chromic acid remaining in the solution is thus proportionate to the quantity of sulphuric acid in the sample. The chromic acid is estimated by means of ferrous chloride and titrated permanganate of potash.

M. Quantin claims priority for the above method. His process is very little different, but he considers it necessary to have solutions of chromate and chloride which are in absolute agreement, and he even gives corrections to use in case the chromate is the stronger.

4. Mohr and Classen have described a process based on the same principle, but the chromic acid is titrated with iodide of potassium and hyposulphite of soda, using starch as an indicator; this process cannot be recommended, as it requires too many reagents.

5. Windisch proposed, for estimating the sulphates in brewery waters, a method of estimation very similar to the one we have just described, but the excess of chromic acid is titrated by means of arsenious acid and iodine. The disadvantage of this process is, that it is almost as long as the gravimetric method.

V. Methods based on Precipitation in the form of Sulphate of Lead.

1. Levöl was the first to propose the use of salts of lead. He poured a titrated solution of lead into the solution containing the sulphates: the end of the operation is shown by the colour assumed by the iodide of potassium which is used as an indicator.

2. A. Guyard, using the same reagents, puts the solution to be titrated into the burette, and runs off the quantity necessary to decolourise a known volume of titrated acetate of lead, coloured with a drop of iodide of potassium. Again, we can precipitate the sulphates with an excess of acetate of lead, and measure the excess with bichromate and salts of silver. None of the methods based on the action of salts of lead can be applied in the presence of chlorides; this is their weak point, for there are many analyses made where we do not detect the chlorine in the state of combination in which it exists in the sample, or where its presence is due to the preliminary solution of the sample.

VI. Method of Félix Marboutin.

We precipitate the sulphuric acid with chloride of barium; the excess of baryta is then precipitated by a quantity of chromate greater than that required by all the chloride of barium used; the chromic acid remaining in the solution is oxidised by a known volume of an arsenious solution; the excess of arsenious acid is measured with titrated iodine.

The reading of iodine obtained in an analysis of water, subtracted from the reading obtained with distilled water free from sulphuric acid, gives the volume of iodine solution equivalent to the sulphuric acid in the sample.

The following is our method of working:—

One hundred c.c. of the water to be analysed are acidulated with HCl, then boiled to drive off carbonic acid; the temperature is then lowered to just below boiling, and 30 c.c. of chloride of barium are added drop by drop; it is then allowed to stand twelve hours at 40°, for the precipitate to become well agglomerated. Neutralise with a few drops of ammonia, and add 30 c.c. of chromate of potassium.

The liquid is gently heated, and then after cooling made up to 300 c.c. 100 c.c. of the clear liquid is taken and 2 c.c. of $\frac{1}{2}$ sulphuric acid, and 5 c.c. of arsenious acid are added; it is then gently warmed and shaken until completely decolourised. To the solution, neutralised with carbonate of potassium, we add from a burette a titrated solution of iodine, with starch as an indicator.

t = the value of 1 c.c. of iodine in m.grms. of I.

n = the number of c.c. of iodine used with 100 c.c. of distilled water.

n_1 = the number of c.c. of iodine used with 100 c.c. of the sample.

x = the number of m.grms. of sulphuric anhydride in the sample.

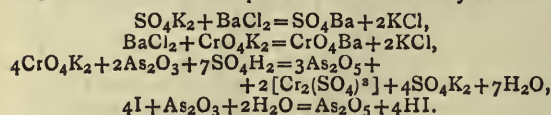
Then—

$$x = 10 (n - n_1) 3\frac{1}{2} \frac{4 \times 80}{3 \times 4 \times 127} \frac{N}{50}$$

If the solution of iodine is exactly $\frac{N}{50}$, that is to say, if 1 c.c. of iodine is equal to 2.54 m.grms. of iodine,

$$x = 16 (n - n_1).$$

The reactions which take place in the above analysis are—



The solutions used are—

Crystallised chloride of barium, 4.8 grms. per litre, $\frac{N}{50}$,

Crystallised chromate of potassium, 3.9 " $\frac{N}{50}$,

Arsenious solution, 4.95 grms. arsenious acid per litre.

Dissolve 4.95 grms. of arsenious acid in water containing 10 grms. of potash, heat gently; after cooling make acid with sulphuric acid, and make up to a litre. Iodide solution: weigh out 2.54 grms. of bi-sublimed iodine, dissolve in water with 5 grms. of iodide of potassium; make up to a litre and titrate with a known weight of hyposulphite of soda.

Our method has several advantages over the others we have mentioned; it has a distinct determination; it does not require *filtrations* or *washings*; only one titrated solution is necessary; it takes cognisance of the possible impurities in the reagents used, by a comparative reading with distilled water. It gives results absolutely comparable with those obtained gravimetrically, and that with a considerable saving of time—of at least three-quarters.

It differs from the method described by Windisch, and other methods based on the use of solutions of chloride of barium and chromate of potassium, in that it is not necessary to use a volume of chromate *exactly* equivalent to the volume of chloride of barium used. We measure, in fact, the difference between the readings made with a water deprived of sulphuric acid and a water containing some. The absolute value of these readings is therefore a matter of indifference.

Our method can be applied to all estimations where sulphuric acid can be isolated by precipitation with chloride of barium. We must, however, point out that in the case of waters containing much organic matter, such as sewage, it is first of all necessary to get rid of this matter, the reading of iodine being in such cases too high, and varying with the nature of the organic matter present. In the case of water from springs, rivers, wells, and surface drainage, the method gives excellent results.

M. Marcel Molimé has compared the figures obtained by our method with those obtained by Windisch's. The difficulty of obtaining solutions of barium, and of chromate, strictly equivalent in the latter case, has led him to determine the corrections necessary from the fact of using solutions not absolutely equivalent.

T_1 being the value of arsenious acid in iodine.

T_2 " " chromate in arsenious acid.

I the reading of iodine in the analysis.

The weight of sulphuric anhydride in Windisch's method is given by—

$$I_2 \frac{T_1 - I}{T_1 \times T_2}$$

We have found that—

1. Five c.c. of arsenious acid decolourises 25.2 c.c. of iodide solution, from which we get $T_1 = 25.2$.
2. Ten c.c. of chromate added to 100 c.c. of water and 10 c.c. of arsenious acid, required 20.6 c.c. of iodine. 10 c.c. of chromium = $(25.2 \times 2) - 20.60$ or 29.80 of iodine.

$$T_2 = \frac{5}{2} \cdot \frac{29.8}{25.2} = 2.956.$$

3. If we use a solution of iodide which is not exactly 50/N, we must correct the readings, so that they do represent a solution of 50/N; in the present case the coefficient by which the readings must be multiplied is—

$$C = \frac{25.0}{25.2} = 0.992.$$

4. From 30 c.c. of chromate + 30 c.c. of baryta, made up to 300 c.c. with distilled water, we take 100 c.c., to which we add 5 c.c. of arsenious acid. The reading shows 24.70 c.c. of iodine. The difference $25.2 - 24.7 = 0.5$ c.c., measures one-third of the value of the difference which exists between the 30 c.c. of chromate and the 30 c.c. of barium. The formula, calling x the correction to be made, is as follows:—

$$\left[(T_1 - I - x) \right] \frac{0.992 \times 12}{\frac{5}{2} \cdot \frac{29.8}{25.2}} = 25.2.$$

$$(T_1 - I - x) \frac{0.992}{29.8} \cdot \frac{2}{5} = 12.$$

$$(25.2 - I - x) = 15.98.$$

RELATIONS CONNECTING THE THERMAL CONSTANTS OF THE ELEMENTS.

By NOEL DEERR.

EMPIRICAL relations existing between the thermal constants of the elements have been proposed by Piéet, Richards, Crompton, and Deerr. As these relations are all very similar, and in some cases identical, it is here intended to collect together—and to some extent to criticise—the work that has been already done.

The following notation is used in this article:—

T = Melting-point absolute in degrees Centigrade.

L = Latent heat of fusion per unit mass.

S = Mean specific heat between -273° and T .

C = Mean coefficient of expansion between -273° and T .

A = Atomic weight.

V = Atomic volume.

W = Valence.

To the various calculations the quantities S and C are generally those determined between 0° and 100° . The relations it is proposed to discuss are—

$$1. TC\sqrt[3]{V} = \text{constant. (Piéet).}$$

$$2. TC = \text{constant. (Deerr, CHEMICAL NEWS, vol. lxxi, p. 303).}$$

$$3. \frac{LC}{S} = \text{constant. (Deerr, CHEMICAL NEWS, vol. lxxi, p. 303).}$$

$$4. \frac{TS}{L} = \text{constant. (Deerr, Proc. Chem. Soc., xvii., 6, 95).}$$

In the relations 2, 3, 4, the constant is stated to hold only between such elements as bear a close relationship to each other, and hence generally for members of any one periodic group.

5. $\frac{AT}{LW} = \text{constant.}$ (Crompton, *Trans. C. S.*, 145, 240).

6. $ALC\sqrt[3]{V} = \text{constant.}$ (Richards, *CHEM. NEWS*, vol. lxxv., p. 278).

7. $\frac{L}{TS} = \frac{1}{2}.$ (Richards, *Journal of the Franklin Institute*, 1893).

Before proceeding to discuss these relations, the writer would draw attention to the widely variant values given by different observers to much of the data in question; the writer has been at great pains to use the most reliable determinations, and in such cases where he has not been able to discriminate between different values, both are given.

Of the relations given above, those numbered 4, 5, 7 are identical, with differences of detail; and the main idea of 1 and 2 is the same; the relation 3 follows from a combination of 2 with 4, precisely as 6 is obtained by Richards by combining 1 with 7, and putting $S.A.S=6.4$.

In discussing the dependency of the latent heat of fusion on the total heat at the melting-point, Crompton, who does not introduce the specific heat, but uses by preference the atomic weight, attempts to reduce the values he obtains to one constant by the introduction of the valency. The introduction of this quantity into the equation is, in the opinion of the writer, a disadvantage; for, besides destroying the dimensions of the equation and making the law almost unintelligible, the values so obtained are *not* constant, varying quite regularly from 0.972 for lead to 1.789 for silver, with a mean value of 1.370; and, further, in the case of bromine and iodine, Crompton has to assume, on very slender grounds, a hypothetical triadic valency, whilst in the case of phosphorus and sulphur he has recourse to the quantity "reciprocal of the valency." Richards, in discussing the relation, gives it a definite physical meaning, stating the latent heat of fusion to bear a constant ratio ($\frac{1}{2}$) to the total heat at the melting-point, but he does not appear to realise the bearing of chemical relationships upon the value of the ratio. Deerr expressed the relation in terms of temperature, naming the quantity $\frac{L}{S}$ the "temperature equivalent of the latent heat of fusion," and he restricted the law as only holding between elements of the same valency.

In Table I. are given values of T, T S, L, and of the ratio $\frac{TS}{L}$.

TABLE I.

Element.	T.	T S.	L.	$\frac{TS}{L}$.
Sodium	365	108.6	32.7	3.27
Potassium	335	56.6	15.7	3.67
Copper	1470	140.	43.0	3.25
Silver	1220	69.5	21.1	3.29
Silver	1220	69.5	24.7	2.81
Thallium	560	17.4	5.1	3.41
Zinc	695	65.0	28.1	2.35
Zinc	695	66.0	22.6	2.92
Cadmium	601	34.2	13.6	2.51
Aluminium	1150	256.	100.	2.56
Mercury	233	7.2	2.8	2.57
Palladium	1700	98.7	36.3	2.72
Gold	1330	42.7	16.3	2.62
Platinum	2300	71.3	27.1	2.60
Gallium	286	21.9	19.2	1.14
Bismuth	535	14.4	12.5	1.15
Lead	603	18.7	5.4	3.47
Tin	503	28.2	14.5	1.90
Bromine	266	27.5	16.2	1.76
Iodine	386	20.8	11.7	1.78
Sulphur	388	69.1	11.8	5.19
Phosphorus	317	59.9	5.0	12.0

Referring to the table, it is seen that the elements Na, K, Cu, Ag, Tl give a constant well within the limits of experimental error. Of these, the first four all belong to the same periodic family; and Tl, although a member of a different family in the thallous compounds, is very similar to the alkaline metals. Following these, there are seven elements giving a value closely approximating to 2.5. Of these, Zn, Cd, Hg are members of one family; Pt, Pd, Au are elements closely allied, although they cannot be strictly said to belong to one family; Al, too, gives a value the same as for these elements, but the data of this element is so uncertain as to render discussion futile. The closeness of the values for the zinc elements and the platinum elements is, in the writer's opinion, accidental, and does not point to an absolute identity. Ga and Bi, although not in the same family, give identical values. These elements are, however, allied; both being trivalent and presenting many analogies. The values given by Br and I require no comment. Following on what has been written above, an identity in the values for Pb and Sn would be expected; the discrepancy is not, however, so startling when the marked non-metallic nature of Sn is remembered. The values for P and S, compared with others, appear abnormal. The specific heat of these elements is obtained near to their melting-point, at which it is at its greatest value; as this quantity varies largely, the cause of the high values may lie therein.

The discussion of this relation can be best concluded by using the law to predict unknown latent heats of fusion. In doing so, the writer was at times in doubt in deciding which of the various values to use; in the table subjoined in such cases the writer has given both values. In the case of In he thinks preference should be given to the value obtained from the constant for Na, &c.; the analogy between In and Tl being so marked. And in the case of arsenic he prefers the value obtained by a comparison with phosphorus.

TABLE II.

Element.	T.	T S.	L (by calculation).	Constant used.
Lithium ..	450	41.0	12.4	3.3 Mean of Na, &c.
Magnesium ..	1020	275.	106.	2.6 Mean of Zn, &c.
Indium ..	450	23.2	7.0	3.3 Mean of Na, &c.
Indium ..	450	23.2	20.4	1.14 Mean of Ga and Bi.
Antimony ..	700	37.1	32.	1.14 Mean of Ga and Bi.
Arsenic ..	685	57.2	50.	1.14 Mean of Ga and Bi.
Arsenic ..	685	57.2	4.8	12.0 Value for P.
Cobalt ..	1700	182.	68.	2.65 Mean of Pt, &c.
Iron ..	1800	198.	75.	2.65 " "
Nickel ..	1900	215.	81.	2.65 " "
Rhodium ..	2000	122.	46.	2.65 " "
Ruthenium ..	2300	143.	53.	2.65 " "
Osmium ..	3000	94.	35.	2.65 " "
Iridium ..	2800	90.	33.	2.65 " "
Selenium ..	490	41.2	8.	5.2 Value for S.
Tellurium ..	653	30.9	6.	5.2 " "
Chlorine ..	200	36.	20.	1.77 Mean of Br and I.

The discussion of the relations $TC\sqrt[3]{V} = \text{constant}$ and of $TC = \text{constant}$ is rendered difficult by reason of various observers obtaining different values for both T and C. In calculating Table III. the writer has not given the different values obtained, but has used those values which he has considered most reliable. In considering the relation, then, a fairly wide margin must be allowed for experimental error. The writer is so placed as not to have access to Pictet's paper, and doubtless some of the values of the thermal constants used by him differ slightly from those used by the writer. In Table III. the values of T are those already given in Table I.

On reference to the values of $TC\sqrt[3]{V}$ it is seen that

TABLE III.

Element.	$C \times 10,000.$	T C.	$T C \sqrt[3]{V}.$	$\frac{LC}{S}.$	$ALC \sqrt[3]{V}.$
Sodium	0.71	0.026	0.074	0.0078	0.151
Potassium	0.84	0.028	0.099	0.0079	0.175
Silver	0.20	0.024	0.052	0.0074—0.0086	0.099—0.116
Copper	0.18	0.027	0.052	0.0081	0.094
Magnesium	0.27	0.028	0.077	—	—
Zinc	0.28	0.019	0.040	0.0067—0.0081	0.086—0.107
Cadmium	0.32	0.019	0.044	0.0081	0.113
Mercury	0.61	0.014	0.034	0.0054	0.081
Aluminium	0.23	0.027	0.059	0.0094	0.136
Thallium	0.31	0.017	0.044	0.0050	0.096
Indium	0.42	0.019	0.047	—	—
Lead	0.27	0.016	0.042	0.0047	0.077
Tin	0.28	0.014	0.035	0.0071	0.120
Phosphorus	0.12	0.0039	0.009	0.0003	0.005
Arsenic	0.056	0.0038	0.009	—	—
Antimony	0.11	0.0077	0.020	—	—
Bismuth	0.14	0.0075	0.021	0.0061	0.102
Sulphur	0.64	0.025	0.063	0.0037	0.060
Selenium	0.37	0.022	0.055	—	—
Tellurium	0.17	0.013	0.036	—	—
Iron	0.11	0.020	0.039	—	—
Cobalt	0.13	0.022	0.041	—	—
Nickel	0.12	0.023	0.043	—	—
Rhodium	0.096	0.019	0.039	—	—
Ruthenium	0.085	0.019	0.040	—	—
Palladium	0.11	0.019	0.039	0.0069	0.094
Osmium	0.066	0.020	0.042	—	—
Iridium	0.070	0.020	0.042	—	—
Platinum	0.088	0.020	0.042	0.0077	0.099
Gold	0.145	0.019	0.041	0.0074	0.102
Bromine	3.5	0.093	0.278	0.053	1.39
Iodine	2.35	0.090	0.268	0.051	1.13

twelve elements give values lying between 0.04 and 0.05, that ten give values lying between 0.03 and 0.06, and that an absolute disagreement is presented by the remaining ten. Now, if the values of $T C \sqrt[3]{V}$ are constant in preference to values of T C, the introduction of the quantity $\sqrt[3]{V}$ should at least tend to correct the values of T C; but it is this quantity which makes the value of $T C \sqrt[3]{V}$ so high for Na and K, and in the other discordant cases its introduction has no effect. Then, again, however much the atomic volume varies, its cube root shows but little variation, and the introduction of this quantity has but seldom a deciding effect, and when it does happen to do so its introduction is hostile to the equation; to say the least, the reaction cannot hold in the case of Na, K, Mg, P, As, Sb, Bi, S, Br, I, and for the remaining elements the agreement demanded by the equation is by no means perfect.

When, however, the relation is put in the form $T C = \text{constant}$, and restricted as holding only between related elements, regularities at once appear; between Na, K, Cu, Ag; between In and Cd; between Br and I; between P and As; between Sb and Bi; and between the elements in the short periods of four in the periodic classification the agreement is all that could be desired. A partial agreement is shown by Tl and In, and by S and Se. The relation fails to hold between Pb and Sn, and also fails in the case of Mg and Hg when compared with Zn and Cd, and in the case of Te compared with S and Se.

A point which is worthy of notice in comparing the relations $T C = \text{constant}$ and $T C \sqrt[3]{V} = \text{constant}$ is that the former has a definite physical meaning which is not possessed by the latter. Expressed in words, this relation signifies that the expansion from -273° to the melting-point is constant for related elements, and so is a function of the atomic weight.

There remain for discussion the relations—

$$\frac{LC}{S} = \text{constant} \quad \text{and} \quad ALC \sqrt[3]{V} = \text{constant}.$$

The dependency of latent heat of fusion upon total heat at the melting-point in connection with chemical relationships has already been shown. It follows, then, that this relation must also hold. In this relation the effect of chemical relationships is not so well shown; most of the values obtained approximating to 0.0075. The rejection of the relation $T C \sqrt[3]{V} = \text{constant}$ implies the rejection of $ALC \sqrt[3]{V} = \text{constant}$; a conclusion borne out by reference to the table.

The relation $T C = \text{constant}$ is not without a theoretical basis, and was obtained by the writer on an argument like this:—At the absolute zero the sum total of the attractions exercised on and by the particles composing a body is at its greatest value. When, by the influence of a rise of temperature, the body has become liquid, no appreciable error is introduced if the attraction is put equal to zero. Using the same notation as before, the heat supplied is $TS + L$. Assuming that the proportion of heat supplied used in overcoming internal attraction is constant, the equation $TS + L = ma$ results; where m is a constant and a represents the total internal attraction at -273° .

On similar grounds, the heat necessary to cause a body to expand through any fraction of its original length is a measure of the internal attraction. Taking the fraction as unity the equation $\frac{S}{C} = na$ results. From these is obtained the relation—

$$\left(T + \frac{L}{S}\right)C = \frac{m}{n};$$

but since, for related elements, the latent heat has been shown to be dependent on the total heat at the melting-

point, it follows that $TC = \text{constant}$ and $\frac{L}{S} = \text{constant}$ for related elements.

The paper can be best concluded by using the relation $\frac{L}{S} = \text{constant}$ to predict unknown latent heats of fusion.

It appears that those obtained from this equation coincide remarkably with those given in Table II.

TABLE IV.

Element.	L (by calculation).	Constant used.
Magnesium	70°	0'0077 Mean of Zn and Cd.
Indium	67°	0'0055 Value for Tl.
Antimony	32°	0'0061 " Bi.
Arsenic	46°	0'0003 " P.
Iron	75°	0'0075 Mean of Pt, Pd, Au.
Cobalt	63°	0'0075 " "
Nickel	66°	0'0075 " "
Rhodium	48°	0'0075 " "
Ruthenium	55°	0'0075 " "
Osmium	35°	0'0075 " "
Iridium	33°	0'0075 " "
Selenium	84°	0'0037 Value for S.

Windsor Forest, West Coast, Demerara.

A THEORY OF THE AURORA BOREALIS.

By GUSTAV WENDT.

WE have here abstracts from and comments on the researches of Halle, Celsius, Hiorten, Canton, and especially Fritz in his work "Das Polarlicht" (1881, Brockhaus).

We may be reminded, as a not unimportant point in establishing the polar light theory, that oxygen is a magnetic—or, more strictly speaking, a paramagnetic—element, that it assumes polarity by the presence of the earth as a magnet permanently present. As Humboldt remarks, "Every atom of oxygen represents a minute magnet, and in the solid or in the liquid state possesses strong magnetic properties." Hence near the magnetic pole the magnetic attraction occasions the descent of paramagnetic matter, especially oxygen or condensed oxygen, and also dust of all kinds, occasionally metallic dust, especially dust of meteoric iron and of nickeliferous nature. If, according to a large series of recent accurate analyses, the air of the mountains and moors of the Scottish highlands generally contain 21 per cent of oxygen, whilst in the large towns, especially in fogs, it sinks to 20.8, and in deep mines to 20.2 per cent, this fact may be explained by the circumstance that, besides the general diffusion, the magnetic attraction is here brought into play. Every large mountain mass possesses, in a larger or smaller degree, the so-called mountain magnetism. The agreeable sensation in lofty yet protected regions is chiefly due to the presence of condensed oxygen, which is continually drawn downwards in consequence of the "mountain magnetism." That the strength of northern lights is directly connected with the activity of the sun must be considered as demonstrated, and is easily explained by the fact that the strength of terrestrial magnetism increases and decreases in proportion to the activity of the sun.

If Fritz demands categorically that a theory of the northern lights should explain why the phenomenon is only manifested by night, he forgets the observations of Humboldt ("Kosmos," iv., 1858, p. 145), that the phenomenon has been observed near the sun on December 3rd, 1827. Humboldt adds, in a note, that the arches of the northern lights were seen near the sun in North America, in Parma, and at London.

Another feature of the northern lights, i. e., the origin of the so-called speared polar light ray ($\lambda = 5567$) has

been explained by Wüllmer (*Poggend. Annalen*, N. F. 388, p. 619).

Hence the northern lights may be regarded as an electrical phenomenon arising when oxygen and other paramagnetic matter is continuously drawn down from the higher regions of the atmosphere, thus setting up electric currents.—*Naturwissenschaftliche Wochenschrift*.

EARLY AMERICAN CHEMICAL SOCIETIES.*

By Prof. H. CARRINGTON BOLTON.

(Concluded from p. 227).

Columbian Chemical Society of Philadelphia (continued).

DR. ADAM SEYBERT (d. 1825) was one of the earliest American chemists to make a series of analyses of the air by eudiometric methods. Having made twenty-seven air analyses during a voyage across the Atlantic, he compared the results with others made on land, and drew the conclusion that the sea exerted purifying power over the air; his paper before the American Philosophical Society bore the date 1797.

Benjamin Silliman is a name so familiar to American chemists as to require no eulogium in this place. At the founding of the Chemical Society he was forty years of age, and had held the chair of chemistry in Yale College for ten years. It should be remembered that he did not begin publishing the *American Journal of Science* until 1818.

Dr. John S. Stringham, professor of chemistry in New York (institution not specified); Dr. Jared Troust (whose name should be written Gerard Troost), lecturer on mineralogy in the Academy of Natural Sciences, Philadelphia, and afterwards professor of chemistry, mineralogy, and geology in Nashville University (1828-50); Lawrence Washington, Esq., of Virginia; and Dr. Caspar Wistar, professor of anatomy in the University of Pennsylvania, with his relative Charles Wistar, complete the roll of home members.

The prominence of medical men on this list is evident, and is easily explained. Before the days of schools of science, and before colleges devoted a portion of their curricula to scientific studies, almost the only training in science received by American youth was in the medical schools. The chairs of natural history and of the physical sciences were almost exclusively held by physicians whose education more nearly qualified them for teaching these branches of knowledge than the graduates of the classical courses customary in all colleges.

To elevate the standard of membership in the Columbian Chemical Society, a number of distinguished foreigners were enrolled. France contributed Adet, Berthollet, Chaptal, Deyeux, Abbé Haüy, Bouillon-Lagrange, Gay-Lussac, Monge, Guyton de Morveau, Parmentier, Pelletier, Sequin, Thénard, and Vauquelin. Great Britain was represented by Sir Joseph Banks, John Dalton, Sir Humphry Davy, John Davy, J. A. de Luc, Hatchett, Dr. William Henry, Sir William Herschel, Dr. John Hope, John Murray, William Nicholson, Dr. G. Pearson, Mr. W. H. Pepys, Dr. Thomas Thomson, Alexander Tilloch, and Dr. William Hyde Wollaston. Spain was represented by Professor Troust of Madrid, and the other countries of Europe had not a single representative. The absence of such eminent names as Richter, Klaproth, Stromeyer, Trommsdorff, and Gehlen, of Germany, as well as of Berzelius, the Swede, presumably indicates that at this early date communications and exchange of courtesies with Germany and Northern Europe was less common than with England and France.

The Columbian Chemical Society of Philadelphia published in 1813 one volume of "Memoirs."† This forms

* Read before the Washington Chemical Society, April 8, 1897. From the *Journal of the American Chemical Society*, August, 1897.

† Copies of the "Memoirs" are found in Philadelphia libraries, and in the private library of the writer.

a book of 221 pages, octavo, and bears the imprint of Isaac Peirce, No. 3, South Fourth Street, Philadelphia. It contains twenty-six essays, by ten writers, on a great variety of topics, original, speculative, and practical.

No less than eight of the papers are from the pen of Dr. Thomas D. Mitchell, and these I proceed to review. Dr. Mitchell's "Remarks on the Phlogistic and Antiphlogistic Systems of Chemistry" opens the volume. In this essay he supports the Lavoisierian theory of combustion, stating that there is "no necessity for the principle of inflammability;" he cites the experiment of Woodhouse, who obtained an inflammable air by heating charcoal with scales of iron, both being free from water, and points out that Cruikshank, of Woolwich, demonstrated that the inflammable gas thus obtained is gaseous oxide of carbon (carbon monoxide), discovered by Priestley in 1799, and combustible although containing no hydrogen. He compares combustion with neutralisation of an acid and base, saying "Inflammation and acidity are effects resulting from the action of relative causes, and not attributable to a single agent or principle."

Dr. Mitchell's second paper, "Remarks on Heat," deals with speculations on latent heat, objecting to this term and to Dr. Black's theories.

In a paper entitled "On Muriatic and Oxy-muriatic Acids," Dr. Mitchell vehemently attacks the views of Sir Humphry Davy as to the non-existence of oxygen in muriatic acid, clinging to the statement of Lavoisier, that all acids contain oxygen. In a section on combustion he remarks "we have incontestable proof that oxygen gas contains light," and he regards combustion as accompanied by the decomposition of oxygen gas.

Dr. Mitchell's fourth paper is of a more practical character, being the "Analysis of Malachite" from Perkioming, Pennsylvania. The result is given as follows:—"120 grains of the green carbonate contained carbonic acid, 30 grains; quartz and siliceous earth, 68 grains; brown oxide of copper, 15 grains; loss in the process, 7 grains."

The specimen was evidently a poor one; no account was taken of the water, and reporting results in percentages was not in vogue.

In some "Remarks on Putrefaction" the same writer discusses the action of antiseptics, and attributes the virtue of nitrate of potash to the increase of cold produced by the muriate of soda.

Dr. Mitchell's "Chemical View of Secretion," and his "Analysis of Professor Coxe's Essay on Combustion and Acidification," are polemical and speculative; in his "Remarks on the Atmosphere" he argues to prove the atmosphere a chemical union of oxygen and nitrogen.

Franklin Bache contributes three essays to the volume. "An Inquiry into What Circumstances Will Warrant us Justly to Reckon any Substance a Principle of a Common Property of Any Set of Bodies," discusses the much disputed question of that day, whether hydrogen as well as oxygen can be an acid-forming principle. His conclusion being, "it may." Bache's second paper is entitled, "An Inquiry Whether Mr. Berthollet was Warranted from Certain Experiments in Framing the Law of Chemical Affinity, that it is directly Proportional to the Quantity of Matter." In this essay the author points out "the probable way in which this great philosopher fell into this great error." In a third paper styled "Thoughts on the expediency of Changing Parts of the Chemical Nomenclature," Mr. Bache proposes the following names: Nitral acid forming nitrotes, nitril acid forming nitrutes, nitrous forming nitrites, and nitric acid forming nitrates, for the several acid-forming oxides of nitrogen. Fortunately his influence was insufficient to inflict these names on chemical language.

Dr. John Manners contributed four papers to the Memoirs. (1) "Experiments and Observations on the Effect of Light on Vegetables and upon the Physiology of Leaves," which abounds in quotations from Darwin's "Botanic Garden." (2) "Analysis of a Mineral Spring

at the Willow Grove," (fourteen miles from Philadelphia). In this the author was assisted by Dr. Mitchell. They report the action of each testing solution on the water, and conclude that the water contains iron and sulphuretted hydrogen, and show the absence of lime, copper, and carbonic acid." (3) "On the Production of Sulphuretted Hydrogen by the action of Black Sulphuric Acid Diluted with Water on Iron Nails." The acid had been blackened by a piece of cork which had fallen in. (4) "Experiments and Observations on Putrefaction." In this paper Dr. Manners tested the influence of carbonic acid, hydrogen, and other gases on putrefying flesh, and also attempted to collect and analyse the gases generated by the same. He concludes that "putrefaction depends on a destruction of the equilibrium of attractions which exist in the elementary principles of which the animal substance is composed in a healthy state, occasioned by the loss of vitality in consequence of which new compositions and decompositions ensue."

Professor Cutbush, President of the Society, wrote "On the Prognostic Signs of the Weather," and "On the Oxyacetite of Iron as a test for the Discovery of Arsenic;" the latter being a good presentation of his discovery, subsequently used as a quantitative method by Kotschoubey.

Mr. Samuel F. Carl, one of the junior members, has two papers containing analyses, the first of the mineral spring at Bordentown, New Jersey, which proved to be a "carbonated chalybeate water," and the second of two specimens referred to him by the society; these proved to be respectively an iron ore and a ferruginous copper ore. The method of reporting results seems very crude to a modern analytical chemist.

Dr. Joel B. Sutherland contributes "Speculations on Lime," in which he claims that if mortar be made with sand containing common salt, the resultant compound gives so much coldness to the mass that during the whole summer vapour is almost incessantly precipitated on the wall with which it is plastered. He also wrote "A Few Remarks on the Nature of the Nervous Influence." Akin to the latter are the "Thoughts on the Principle of Excitability," by George Ferdidand Lehman, who also wrote "On the Emission of Oxygen by Plants."

Mr. William Hembel, Jr., has two papers, one entitled "Observations on the Formation of Muriate of Potash in the Process of Preparing the Hyperoxymuriate of Potash," which is complicated by the belief that hydrochloric acid is an oxygen compound; and another entitled "A New Method of Mounting Woulfe's Apparatus," which is unintelligible owing to the omission of a woodcut to which the text refers.

Mr. Edward Brux, of France, one of the junior members, writes "Upon the Effects of Various Gases upon the Living Animal Body," which consists largely of speculations: notwithstanding which he cites an admirable passage from Dr. Bostock; "Physiologists have, in general, been more inclined to form hypotheses than to execute experiments, and it has necessarily ensued from this unfortunate propensity that their science has advanced more slowly than perhaps any other department of natural philosophy." Unfortunately this truth was not fully recognised by the members of the Columbian Chemical Society.

A contemporary journal (N. Y. Medical Repository), in reviewing the "Memoirs," uses the following quaint language: "It is highly gratifying to behold a band of worthies like those before us, laboring to analyse the compounds which they find ready made, to form by synthesis new combinations in the laboratory, and thereby to deduce correct doctrines from the facts which are disclosed. We cordially congratulate them on their noble occupation and on the progress they have made. We hope they will be persevering and undaunted. And if from this beginning there shall arise great improvements in theoretical disquisition, as well as in economical exercise, we shall rejoice with a mingled glow of amicable and patriotic sentiment."

III. The Delaware Chemical and Geological Society.

The Delaware Chemical and Geological Society was organized at Delhi, Delaware County, New York, September 6, 1821; the first meeting was held at the hotel of G. H. Edgerton in the village, and the following officers were chosen:

President—Charles A. Foote.
Vice-President—Rev. James P. F. Clark.
Recording Secretary—Charles Hathaway.
Corresponding Secretary—Dr. Calvin Howard.
Treasurer—Selah R. Hobbie.
Directors—Cornelius R. Fitch, R. W. Stockton, Ebenezer Steele.

The society was composed of "between forty and fifty well-informed and respectable inhabitants" of the County of Delaware. The following gentlemen were elected corresponding members: Colonel Henry Leavenworth, U.S.A.; Edwin Crosswell, of Catskill, and O. Rice, of Troy.

The society had for its object the improvement of the members in literature and science, especially in chemistry and mineralogy. The members planned to form a library and to secure a chemical laboratory; they made a collection of the minerals and rocks of the region, which was still preserved in the Delaware Academy in 1856. The meetings of the society were held quarterly, and at each an essay or an "original scientific discourse" was presented; it was, however, not long sustained.

In reviewing the condition of chemical science in the United States, as indicated by the membership and achievements of these early societies, we note that those who held the most prominent places were handicapped by the necessity of devoting a large part of their intellectual energy to topics quite outside of the domain of chemistry itself. The active members were either busy with the art of healing, or with teaching several branches of the physical and natural sciences, and too often chemistry was regarded in the colleges as a kind of side issue, or appendix to the more important subjects of instruction. This was caused by the necessity of earning a competence at a time when there was no opportunity of reaping pecuniary rewards by skill as an analyst, or by the application of science to the manufacture of products involving chemical knowledge. Indeed, in default of this stimulus to laboratory work, it is not surprising that the papers read to the societies were largely either reviews of the grand discoveries made by Europeans, or essays in which the imaginative faculty was given free play, it being far easier to indulge in speculation than to discover new facts.

In the early struggles of a country to secure a place among nations, few men of ability can devote their energies to the pursuit of science for science's sake; the environment is more favourable to development of the inventive faculty than of the peculiar talent for conducting abstruse researches in an exact science. Add to this the limited facilities for acquiring chemical knowledge in the New World, and the distance of amateurs from the European head-centres of learning, and it is certainly noteworthy that American chemists combined to form associations for mutual improvement and the advancement of their calling at so early a period.

A fourth attempt to establish a chemical society was made at New York City in 1876; the organisation was at first somewhat restricted in its plan, but in 1892 a change in its constitution was effected, which broadened its scope, and it now forms a strong, influential, and truly national society. Its 1106 members, working in nine chartered sections, represent forty-seven states and territories, besides several countries of Europe, South America, and distant Australia. Its Journal, comprising 1150 pages annually, is an authoritative medium for the preservation and diffusion of the researches made in the United States, and its annual meetings, held in diverse localities, strengthen the bonds which unite its members in good fellow-

ship, and in the pursuit of their common profession. *Long may the American Chemical Society continue in its prosperous career!*

PROCESS FOR SEPARATING AND DISTILLING BROMINE FROM A MIXTURE OF ALKALINE CHLORIDE AND BROMIDE.

By H. BAUBIGNY and P. RIVALS.

THE decomposition of bromides by the action of a solution of copper sulphate and potassium permanganate does not permit us to collect the bromine or to determine it directly by diffusing when operating in a vacuum. It partly attacks the grease which serves as a luting. We can therefore know the weight, having previously determined that of the chlorine and the sum of the weights of the two elements. But every process of indirect determination is imperfect, especially when the body in question is the smaller in quantity.

We have therefore sought to remove mechanically the bromine from the liquid, either by ebullition or by a current of air.

The method of ebullition in which the watery vapour formed has the object of expelling the free bromine has also revealed the inconveniences involved. The first is the formation of enormous volumes of condensed water. Secondly, the ebullition causes rapid variations of the liquid volumes. Now the alkaline chlorides, stable at 100°, if the solution is not too strongly charged with salts of copper, are decomposed if the concentration exceeds certain limits. The use of a current of air, on the contrary, has permitted the easy solution of the question, and can be used at any temperature. — *Comptes Rendus*, No. 15, October 11, 1897.

NOTICES OF BOOKS.

Annali del Laboratorio Chimico Centrale delle Gabelle, pubblicati da Vittorio Villavecchia. Vol. III. Roma, 1897. 242 pp. 8vo.

The first volume of the Annals of the Central Chemical Laboratory of the Customs was issued in 1891, and covered the period from 1886-89; the second volume bore the date 1893; the present volume chronicles the work done in the years 1890-96. More than four thousand eight hundred analyses were made in the year 1896, the total number for the period 1890-96 being 24,913. The substances examined included wines, vermouth, beer, spirits; fixed, mineral, and volatile oils; mineral waters; coffee, chocolate, spices, confectioners' preserves, candy, &c.; condensed milk; effervescent magnesium citrate, salts of Stasfurt; vegetable and mineral colouring substances, varnishes, inks, minerals, cements, cereals; fats, soaps, waxes; and a variety of chemical and pharmaceutical products.

The volume contains records of the following researches:—

I. "On the Substance found in Sesame Oil, and on the Relation which it bears to the characteristic Colour Reaction of this Oil." By V. Villavecchia and G. Fabris.

II. "On the Composition of Italian Flour." By G. Fabris and O. Severini.

III. "On the Paper Mulberry." By M. Tortelli.

IV. "Soap Analysis." By R. Moreschini.

V. "On Illuminating Oil and its Consummation in Italy." By A. Volpi and R. Ruggieri.

VI. "On Dulcina, or Paraphenetol Carbamide, and a Method of Recognising it." By R. Ruggieri.

VII. "Analysis of Candies." By A. Bianchi and O. Severini.

VIII. "On the Chemical Composition of some Greek Wines." By R. Moreschini.

IX. "Researches on *Dé gras* (Fish Oil)." By M. Tortelli.

X. "On the Estimation of Glycerin in Sweet Wines." By G. Fabris.

The volume is well printed, but the absence of running-headlines makes it difficult to find the several essays.

The monograph on the paper-mulberry is accompanied by a plate.

H. C. B.

CORRESPONDENCE.

ARGON AND HELIUM.

To the Editor of the Chemical News.

SIR,—I would like to call the attention of chemists who are working at the gases argon and helium to the fact that there is near Mallow, Co. Cork, a mineral spring which evolves large quantities of nitrogen. According to an analysis by Prof. Daubeny the constitution of the evolved gas is—

Nitrogen	93.5 parts
Oxygen	6.5 "

100.0

Near Hillsbrook, in the parish of Killrerin (Barony of Clare), is another spring, of which the following analysis of the evolved gas was made:—

Analysis of a Wine Pint of Water in May, 1842.

Nitrogen	0.59 cub. ins.
Carbon dioxide	1.0 "

Most probably the above-mentioned gases contain argon or even helium.—I am, &c.,

C. P. FINN.

The Yorkshire College, Leeds,
November 1, 1897.

THORIUM ACETYL-ACETONATE.

To the Editor of the Chemical News.

SIR,—If my request is not unreasonable I should feel extremely favoured if anyone could inform me whether the acetylacetone referred to in the article on "Thorium," which appeared in No. 1971 of the CHEMICAL NEWS (vol. lxxvi., p. 110), is the ordinary acetone or no.

If it is not acetone I am at a loss to understand to what the author refers, and am unable to find any information on the subject either in "Dict. Chem." Thorpe and Muir, or Gmelin or Watts.

Presuming acetone to be meant by acetylacetone I have carefully followed the process, but failed to prepare the acetylacetone of thorium mentioned therein.

Taking an exceptionally pure thoria salt, which was prepared for a special purpose by repeated precipitations, as "hypo," "oxalate," and ultimately re-crystallising the double ammonium salt several times, I converted it into the chloride, precipitated with ammonia, washed, and removed excess of water by pressure.

This purified hydrate was suspended in dilute alcohol (1 ab. to 5 aq.), mixed with acetone, and evaporated to dryness on water-bath. The process failed, however, to yield any substance soluble in chloroform, nor were repeated trials more successful.

As the commencement of paragraph 2 is slightly ambiguous I tried a further experiment by acting on the $\text{Th}(\text{HO})_4$ with peroxide of hydrogen before the acetone treatment, after which I carefully followed the process as given by M. Urbain. Obtaining no better results I am

forced to conclude that acetylacetone is some unusual substance beyond the knowledge of an ordinary inorganic chemist, and have therefore ventured to trouble you in this matter.—I am, &c.,

ALLANITE.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 16, October 18, 1897.

Impurities of Crude Copper.—M. Schlagdenhauffen. —The treatment by nitric acid of the sublimate in one of the author's tubes yields on analysis all the characters of selenious acid.

Electrolytic Conductivity of Trichloroacetic Acid.—Paul Rivals. —Not adapted for useful abstraction.

The Mean Molecular Weight of the Soluble Matter in Seeds during Germination.—M. Maquenne.

No. 17, October 26,

Novel Procedure for obtaining Instantaneous Radiographs.—G. Séigny. —Following the indications of Dr. Max Lévy the author takes a very thin plate of glass, which he coats on both sides with silver gelatino-bromide and then allows it to dry in this emulsion. He prepares meantime two flexible screens of cloth with violet calcium suspended in celluloid. When the screens are dry they are applied to each side of the double emulsion plate, the whole being then placed in a frame exerting a pressure on the surfaces by means of two plates of cardboard, and proceed as usual.

A New Bianodic Vessel for Red Phosphorescence. G. Séigny and E. Gundelag. —The authors have prepared the glass for these vessels by incorporating with coloured glass transparent and not fluorescent powdered albumen and calcium carbonate, or preferably didymium chloride. The glass thus prepared has the following properties:—1. Its fluorescence is in red and not green. 2. It emits twice as many X rays as the ordinary glass. 3. The fluorescence which it excites upon the screen is more brilliant and of yellowish green colour.

Researches on Saline Solutions. Lithium Chloride. —Georges Lemoine.

On Basic Magnesium Salts.—M. Tassilly. —The researches of various *savants* on magnesium oxychlorides and the author's own on the oxybromide tend to establish an approximation between this metal and zinc as regards the basic salts.

Separation and Direct Determination of the Chlorine and Bromine contained in a Mixture of Alkaline Salts.—H. Baubigny and P. Rivals.

Some Compounds of Metallic Acetates with Phenylhydrazine.—J. Moitessier. —Phenylhydrazine forms with metallic acetate of the magnesian series compounds analogous to the phenylhydrazinic chlorides, bromides, iodides, and nitrates. The author has prepared the zinc, cadmium, manganese, cobalt, and nickel phenylhydrazines by heating in the water-bath a mixture of phenylhydrazine in an alcoholic solution and of a pulverised metallic acetate.

Methods of Determining Diabetic Sugar.—F. Landolph. —1. The polaristrobometer alone indicates the real quantity of active diabetic sugar. 2. The coefficient of reduction gives double and even threefold the polaristrobometric sugar. 3. Fermentation shows a very vari-

able quantity of sugar according to the duration of the fermentation.

Optical and Reductive Power of the Flesh of Flies.—F. Landolph.—The author finds that a filtered solution of the flesh of flies obtained by trituration with cold water is opalescent and strongly levo-rotatory. Its reductive power is certainly greater than that of diabetic sugar. The reductive power of the aqueous extract of spiders is also very high.

Journal de Pharmacie et de Chimie,
Series 6, vol vi., No. 7.

On the Question of Matches. Phosphorism.—A. Riche.—A long paper, to be continued, not suitable for abstraction.

Detection of Acetone in Urine.—A. Mallat.—In 1896 the author published the method of detecting acetone in urine, which consists of precipitating 100 c.c. of urine with 10 c.c. of subacetate of lead, then distilling a portion of this liquid with caustic soda, then adding to a small portion a solution of iodine and iodide of potassium. On reversing the test-tube once, the presence of iodoform is shown by the smell and the cloudy appearance of the solution, indicating that acetone was present in the urine. M. Bretet, who has used this method, has modified it in the following manner:—He does not treat the urine with subacetate of lead, but he distills it in the presence of tartaric acid; he stops the operation when about one-tenth has been distilled over. Then, in two test-tubes, he makes the mixture of caustic soda and of iodine in iodide of potassium, and thus obtains an absolutely clear and limpid reagent. To one of these tubes he adds the distilled urine under examination, and the second tube he uses as a check. The slightest formation of iodoform causes a cloudiness in the tube, and denotes the presence of the smallest traces of acetone.

Composition of Potatoes.—M. Balland.—The author finds that 3 kilogms. of potatoes before or after cooking, equal to about 1200 grms. of fried potatoes or 700 grms. of desiccated potatoes, contain the same amount of nitrogenous and amylaceous matters as 1 kilogram. of ordinary white bread.

Assay of Alloys of Copper and Nickel.—A. Riche.—The use of coins made of an alloy of copper and nickel in two of the French colonies, has rendered necessary at the Mint a rapid and exact method for the analysis of these alloys. They are not of the same composition: one containing 25 per cent of nickel, and the other only 15 per cent. The alloys being binary, it is only really necessary to determine one of the metals—the copper, for instance. But as the nickel is the most expensive of the two metals, it is preferable to estimate it directly rather than to deduce its weight by difference. The operation is carried out as follows:—About 1 grm. is attacked with the smallest quantity possible of nitric acid on a sand-bath; when finished, add a little water containing 5 or 6 drops of sulphuric acid and evaporate to dryness. Take up with water still containing a little sulphuric acid, and again evaporate to dryness to make sure that all the nitric acid is driven off; re-dissolve in water with a little sulphuric acid, and pour into an electrolytic crucible, filling it about one-third full. The copper alone is deposited. The remaining solution is saturated with ammonia and electrolysed with three Daniell cells. This brings down the nickel, and the whole process gives very concordant results. The difference between the total weights found and 100 represents the impurities—consisting of sesquioxide of iron, also oxides of manganese and aluminium—in the nickel. In the case of a bronze coin, 475 m.grms. of copper were deposited in four hours with a current of half an ampère.

No. 8.

On Antimonic Acids and Antimonates.—M. Delacroix.—Frémy described two antimonie acids and

their salts, but many chemists will not admit the existence of more than one. M. Delacroix has taken up the study and is convinced that there are really two: pyroantimonic acid, giving acid pyroantimonates and neutral salts; and orthoantimonic acid, giving acid, neutral, and basic salts. The solution of pyroantimonic acid was prepared from the hydrate by pouring SbCl_3 into 20 or 25 times its weight of cold water, or by treating SbCl_3 (previously dissolved in a little water), in the same manner, then saturating with chlorine. The excess of chlorine is driven off by a current of air directed on to the mixture. After standing an hour or two, the hydrate is transferred to a filter, washed a little, and pressed strongly between blotting-paper. It retains a little HCl very tenaciously, but not more than if it had been washed for a much longer time. The hydrate is then placed in cold water, in which it slowly dissolves. The solution, which is colourless, is evaporated to dryness and calcined, forming Sb_2O_4 , from the weight of which the amount of anhydride is calculated. Pyroantimonic acid, when warmed for some time at 100° , becomes transformed into the more basic orthoantimonic acid. The same transformation takes place spontaneously at the ordinary temperature in a few days. The solutions thus prepared are slightly opalescent. The author intends studying the basic orthoantimonates and the neutral pyroantimonates, which are new salts.

MISCELLANEOUS.

The Commercial Development Corporation, Limited.—We are officially informed that letters of allotment and regret of the above Company have been posted.

Society of Arts.—The Society of Arts will commence its Session (the 144th from its foundation in 1754) on the 17th inst. with an address on "The Colonies: their Arts, Manufactures, and Commerce," by Major-General Sir Owen Tudor Burne, G.C.I.E., K.C.S.I., the Chairman of the Society's Council. There will be four meetings on successive Wednesdays before Christmas, at which papers will be read by Prof. James Douglas, on "The Progress of Metallurgy and Metal Mining in America during the last Half Century"; by Prof. Leonard Waldo, D.Sc., on "The American Bicycle—the Theory and Practice of its Making"; by Bennett H. Brough, on "The Stockholm Exhibition of 1897"; by Samuel Rideal, D.Sc., on "The Purification of Sewage by Bacteria." The first course of Cantor lectures will consist of three lectures, to be given on Monday evenings, commencing on the 29th inst., by Dr. Eugene F. A. Obach, F.C.S., on "Gutta Percha."

Imperial Institute.—A commercial reading-room, open free to the general public, has been established at the Institute, in the hope that it may be specially useful to mercantile men, manufacturers, &c., either resident in, or visiting the Metropolis. A considerable number of commercial and technical publications (British, Indian, and Colonial and Foreign) can be read or consulted in the Reading-room. In addition, the room will contain a trade circulars, information in regard to shipping, transit by rail, &c. Maps of different portions of the Colonies are likewise exhibited, and many maps or charts included in the collection provided in the Map-room of the Institute may be consulted on application. Works connected with the Colonies and India (such as Diplomatic and Foreign Consular Reports, Directories, Year-books and Handbooks, Government Gazettes, Market Reports, Prices Current and Statistics, Parliamentary Papers and Blue-books, Statistical Registers, Weather Reports, &c.) are available to the public visiting the Reading-room, on filling up application forms.—*Journal of the Society of Arts.*

NOTES AND QUERIES.

*. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Soluble Essences.—Monazite.—1. Would anyone oblige me by informing me of the best manual on the distillation and manufacture of soluble essences, such as are used in the manufacture of aerated waters. 2. The appearance, &c., of monazite, and where specimens may be procured and at what cost.—C. F. F.

MEETINGS FOR THE WEEK.

WEDNESDAY, 17th.—Sanitary Institute, 8. (See p. 231).
Society of Arts, 8. Opening Address of Session by Major-General Sir Owen Tudor Burne, on "The Colonies—Their Arts, Manufactures, and Commerce."

THURSDAY, 18th.—Chemical, 8. "Decomposition of Camphoric Acid by Fusion with Potash or Soda," by A. W. Crossley, M.Sc., Ph.D., and W. H. Perkin, jun., F.R.S. "Experiments on the Synthesis of Camphoric Acid," by W. H. Bentley, B.Sc., and W. H. Perkin, jun., F.R.S. "Action of Magnesium on Cupric Sulphate Solution," by Frank Clowes, D.Sc., and R. M. Caven, B.Sc. "Properties and Relationships of Dihydroxy-Tartaric Acid," by H. J. Horstman Fenton, M.A.

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CIVIL SERVICE COMMISSION.

FORTHCOMING EXAMINATION.

Dispenser in H.M. Naval Hospitals at Home and Abroad (20 to 25), 17th December. The date specified is the latest at which applications can be received. They must be made on Forms to be obtained with particulars from the Secretary, Civil Service Commission, London, S.W.

Wanted immediately, a Manager for Colour Works, experienced in the manufacture of Red Oxides and Paints.—Write, stating age, experience, and salary required, to "Paint," care of Mr. Tomlin, 62, Ludgate Hill, London, E.C.

THE CHEMICAL NEWS.

VOL. LXXVI., No. 1982.

ON THE ESTIMATION OF COPPER AS IODIDE.

By M. WILLENZ.

THE volumetric estimation of copper as iodide, though old and well known, is being gradually abandoned, not having given, according to a great number of writers, such satisfactory results as were expected. For the last few years, however, chemists have again taken up the study of this question, and the estimation of copper as iodide is being revived, and is even officially adopted in some countries, notably the United States of America.

This method is, in fact, simple and rapid, and only requires the use of ordinary reagents. We were desirous of testing the accuracy and value of the process in question, and we now submit our results to the readers of the *Review*.

We have attempted to apply this method to cupreous pyrites, minerals containing at the most 6 per cent of copper, and we are able to state that the results obtained by following the plan we are about to describe have been very favourable, provided always that certain precautions are taken, and that the conditions of working are always identical.

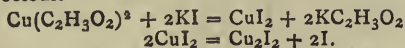
We proceed as follows:—

A solution of hyposulphite of soda is prepared, containing 9.92 grms. of the crystallised salt ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{Aq}$) per litre; this makes a N/25 solution, of which each c.c. is equivalent to 0.00252 grm. of copper. But as the hyposulphite is hardly ever pure, it is absolutely indispensable to control the titration by means of metallic copper, and further, as the hyposulphite alters more or less rapidly, and as its titration value is always going back, it becomes necessary to verify it before each series of experiments, and for this purpose it is well to keep a standard cupric solution.

To prepare this solution we dissolve 10 grms. of electrolytic copper in a mixture of equal volumes of water and nitric acid, $D=1.4$. When all the copper is dissolved, we boil for a long time until all the nitrous vapours are driven off, let cool, and make up to 1 litre.

To determine the titration value of the hyposulphite, we take 10 c.c. = 0.1 grm. of the cupric solution, in an Erlenmeyer flask of 200 c.c. capacity, having a mark at 50 c.c.; add dilute ammonia drop by drop until the blue precipitate of the basic salt no longer forms, then add dilute acetic acid drop by drop to re-dissolve the basic salt, and finally make the solution strongly acid with 5 or 6 c.c. of concentrated acetic acid; dilute to 50 c.c., and add 10 c.c. of a 10 per cent solution of potassic iodide, free from iodate, = 1 grm. KI.

The addition of potassic iodide causes the formation of cupric iodide, which immediately splits up into cupreous iodide and free iodine; the latter gives the solution a brown colour.



The result of this is that 63 parts of copper require 332 parts of iodide of potassium, or, in other terms, 0.1 grm. of copper requires 0.527 grm. of iodide; but as the reaction occurs but slowly when we take only the theoretical quantity, it is better to add 0.1 grm. of iodide, especially as an excess, even when considerable, has no interfering influence on the final results. We allow the iodide to react for two minutes, but no longer, and titrate with the hyposulphite, which is run in until the liquid

takes a clear yellowish brown tint; we then add a little starch solution, and while agitating we add more hyposulphite. As soon as the colour of the iodide of starch begins to change to a dirty greyish violet, we add the hyposulphite by only a drop at a time, shaking thoroughly after each addition.

We have remarked, in fact, that the violet tint will still persist even after all the iodine has been transformed into sodic iodide, but violent agitation will make it disappear, and the straw colour, due to cuprous iodide, shows itself, thus indicating the end of the titration. To get concordant results it is necessary always to stop at the same point; this is not difficult after a little practice.

When carried out in this manner the method gives perfectly concordant and very satisfactory results. We have never noticed the re-appearance of the blue colour, even after standing several hours.

We give below several titrations made with pure cupric sulphate, $\text{CuSO}_4 \cdot 5\text{aq.}$, containing 25.421 per cent of metallic copper. The aqueous solution of the salt was acidulated with 0.5 c.c. of nitric acid, $D=1.4$, then treated with ammonia and acetic acid as above, diluted to 50 c.c.; 10 c.c. of 10 per cent iodide of potassium was then added, the whole let stand for two minutes, and then titrated,—

1 c.c. $\text{Na}_2\text{S}_2\text{O}_3 = 0.00249376$ grm. Cu.

Application of the Method to the Estimation of Copper in Pyrites.—Ten grms. of finely powdered and dried pyrites are placed in a tall narrow beaker, 8 c.c. of water and 2 c.c. of concentrated sulphuric acid are added. Cover the beaker with a watch-glass, and add nitric acid, $D=1.4$, in small quantities, until there is no longer any effervescence; this will require about 25 to 30 c.c. of HNO_3 , then add 3 c.c. more of H_2SO_4 . Boil well, over a naked flame for preference, and after a few minutes take off the watch-glass, and, without washing it, place it temporarily on one side. Continue boiling, and at the same time keep the beaker constantly turning—this is an indispensable precaution—until the mass, becoming thicker and thicker, will no longer run, but becomes quite pasty. Treat it now with hot water and replace the watch-glass, boil for a short time and allow to cool: make up to half a litre and filter.

The residue, which is quite free from copper if the operation has been properly conducted, consists of silica, sulphur, PbSO_4 , ($\text{BaSO}_4, \text{CaSO}_4$). This operation requires a certain amount of skill, and special care must be taken lest the sulphur set at liberty envelopes the unattacked particles of mineral, and thus prevents them being acted upon, but with a little practice this is easily avoided; the operation requires but half an hour at the most.

Take 100 c.c. = 2 grms. of pyrites, of the sulphuric solution thus obtained, in a conical flask, and add a few c.c. more of H_2SO_4 ; boil, and without stopping the heat add, little by little, a warm concentrated solution of hyposulphite of soda.

The addition of the first few drops produces no change, but there is soon a lively disengagement of sulphurous acid gas, and the liquid becomes cloudy, then successively greenish, brownish, chocolate-brown, and finally black, without any precipitate appearing. But by keeping up the boiling the precipitate comes down, agglomerates, and falls to the bottom of the flask, while the supernatant liquid becomes limpid.

This precipitate consists of sulphides of copper, arsenic, antimony, and tin, mixed with free sulphur, but it must be remembered that the last three sulphides are only partially precipitated when not in the presence of a great excess of acid; it may even happen that they are not precipitated at all if the bases are present in small quantities only. The other bases of the copper group are not precipitated,* the iron is reduced to the state of ferrous

* It is, however, worthy of remark that bismuth is precipitated under these conditions, but we have never come across this metal in pyrites.

Grms. CuSO ₄ 5aq.	Cu calculated.	C.c. of Na ₂ S ₂ O ₃ used.			Cu found.	Difference.
		I.	II.	Mean.		
0'10	0'025421	10'15	10'20	10'18	0'025386	-0'000035
0'15	0'038131	15'35	15'30	15'33	0'038229	+0'000098
0'20	0'050842	20'40	20'40	20'40	0'050873	+0'000031
0'25	0'063552	25'50	25'50	25'50	0'063591	+0'000038
0'30	0'076263	30'55	30'60	30'58	0'076259	-0'000004
0'35	0'088973	35'65	35'65	35'65	0'088902	-0'000071
0'40	0'101684	40'75	40'70	40'73	0'101571	-0'000113
0'45	0'114394	45'85	45'90	45'87	0'114389	-0'000005
0'50	0'127105	51'05	51'00	51'02	0'127232	+0'000127
0'60	0'152526	61'20	61'20	61'20	0'152618	+0'000092
0'70	0'177947	71'45	71'40	71'42	0'178104	+0'000157
0'80	0'203368	81'50	81'50	81'50	0'203241	-0'000127
0'90	0'228789	91'90	92'00	91'95	0'229301	+0'000512
1'00	0'254210	102'20	102'20	102'20	0'254862	+0'000652

salt; the aluminium, zinc, manganese, nickel, and cobalt remain in solution.

The precipitate is washed several times with boiling water, first by decantation, and afterwards on the filter. When obtained under the above-mentioned conditions it oxidises very slowly, and may be washed with pure water without any risk, but the washing should be done rapidly. When the wash waters no longer precipitate BaCl₂, we take the filter and its contents out of the funnel, squeeze it gently between blotting-paper to get rid of most of the moisture, then place it, point uppermost, in a porcelain crucible. Heat very gradually, and when all the water is driven off increase the temperature, and finally roast, so that all the sulphur is burnt, and the arsenic, antimony, and tin volatilised; there then remains in the crucible nothing but a mixture of oxide and sulphide of copper.

This is dissolved in 1 c.c. of a mixture of equal volumes of water and nitric acid, D=1.4, and boiled to completely drive off all nitrous fumes; transfer to a 200 c.c. Erlenmeyer flask, with a mark at 50 c.c.; add dilute ammonia, drop by drop, until the precipitate of the basic salt is no longer formed; this is re-dissolved in weak acetic acid, acidulated strongly with 5 or 6 c.c. of strong acetic acid; 10 c.c. of iodide of potassium are then added, and the whole left for two minutes; it is then titrated with hyposulphite, observing the precautions mentioned above.

As we have already said, this method gives very satisfactory results; it is easy to perform, and requires but very little time. Examples:—

1 c.c. Na₂S₂O₃ = 0'0025 grm. Cu.

Grm. Cu.	Grm. As.	C.c. of Na ₂ S ₂ O ₃ .	Cu found.	Difference.
0'0972	+0'4013	39'0	0'0975	+0'0003
0'0647	+0'4735	26'0	0'0650	+0'0003
0'0892	+0'7102	35'75	0'08937	+0'00017
0'1241	+0'8284	49'8	0'1245	+0'0004
0'2004	+1'4672	80'25	0'20062	+0'00022

Pyrites.	Contents of As, per cent.	Cu found	
		By Fresenius's method, per cent.	By this method, per cent.
I.	0'320	3'267	3'272
II.	0'024	0'069	0'081*
III.	0'062	4'584	4'601
IV.	0'182	0'074	0'081*
V.	traces	1'823	1'872
VI.	0'307	5'749	5'771
VII.	Not determined	2'218	2'330

* Five grms. of material used.

It is evident that this method can be applied equally to copper ores properly speaking, to bronzes, alloys, &c., but in these cases the quantity taken for assay must be less, and the solution of hyposulphite used may be more concentrated, a decinormal solution for instance; however

we prefer a weak solution, the inherent errors being then reduced to a minimum.—*Revue de Chem. Analytique*, Vol. v., No. 18.

ON THE DISSOCIATION SPECTRA OF MELTED SALTS.

ALKALINE METALS: SODIUM, LITHIUM, POTASSIUM.

By A. DE GRAMONT.

THE easiest dissociation spectra to study with the condenser spark are those of the salts of the alkaline metals, on account of their fusibility, stability, and simplicity.

To do this, it suffices to deduct from the total spectrum the metallic lines, to get the complete spectrum of the metalloid without having recourse to Plücker or Salet tubes. I therefore preferred commencing with the spectra of the common alkaline metals as they appear in the dissociation of their fused salts. They differ to a certain extent from those yet obtained, either with the pure metal and not much condensation, or with the fused salt and the coil only. Many of the lines, especially those in the most refrangible part of the spectrum, become wider and more diffused; the component parts of double lines being difficult to separate, no matter what dispersion is used, and appearing as fairly wide bands.

Most precise measurements of the wave-lengths of sodium will be found in the paper by MM. Eder and Valenta (*Deutschr. Kais. Akad. d. Wissenschaft*, vol. lxi., Vienna, 1894), who worked with the metal itself in an atmosphere of hydrogen and with a small condenser.

The spectrum of lithium has been measured with great accuracy by MM. Kayser and Runge (*Abhandlung Berliner Akad.*, 1890) in the arc, where the lines are the same as those now given and described.

I have established the lines peculiar to each metal under the following conditions:—1. By subtracting from the complete spectrum of a salt the lines of the combined metalloid, and repeating the experiment with several salts of the same metal. 2. By observing the spectrum of a carbonate, where the carbon lines appear only under special conditions. This has been recently proved (*Comptes Rendus*, vol. cxv., July 19 and 26, 1897). The dissociation of the salt is more or less easy to effect according to its nature; it is easy with chlorides, bromides, iodides, sulphides, sulphates, phosphates, &c.; it is more difficult with carbonates and fluorides, which require a much higher temperature and a greater difference of potential. The measurements of the wave-lengths here given were obtained with a direct-vision spectroscopy with two compound prisms, easily doubling the D line of sodium (Na α), which appeared to be one division of the micrometer apart; these divisions, with a little practice,

could be read to one-tenth. These values were compared with the normal solar spectrum photographed by Rowland. I have kept the alphabetical description for Na and K given by M. Lecoq de Boisbaudran in Plate V. of his "Atlas of Luminous Spectra."

Sodium.

δ	616.1	Strong, bright.
	615.5	" "
α	589.6	Intense.
	589.0	" "
β	568.8	Strong, bright.
	568.3	" "
	567.5	Faint.
	567.0	" "
ϵ	515.5	Fairly strong.
	515.1	" "
γ	498.3	Easily seen, very diffuse, almost joined.
	497.9	
	467.2	Faint, diffused.
	467.0	Fairly bright, diffused.
	454.5	Faint, diffused.
	449.9	Fairly bright, diffused.

Practically, and above all in the presence of metalloids, the spectrum of sodium is reduced to three double lines, bright and characteristic, Na δ , Na α (D of Fraunhofer), and Na β , each one being seen as a single line in a monoprismatic instrument with a medium slit. Na ϵ comes next in strength. The value I give it appears rather high (MM. Kayser and Runge give 515.37 and 514.92). The remaining double lines are much weaker and indistinct, almost forming a single diffuse band. Those marked * could not be split up on account of the condenser used.

Lithium.

	670.8	Strong, bright.
	610.3	Very strong.
	497.2	Strong.
	460.3	Strong, wide, diffuse.
	427.3	Well marked.
	413.2	Well marked, very diffuse.

A spectrum of such simple character, of six lines only, and so easily seen, renders the use of lithium salts particularly favourable for the study of the spectra of metalloids. The first four lines are always seen with the disruption discharge, either on the metal or on the solution of one of its salts, but the relative intensities of the lines vary in the two cases. 427.3 has never been seen before except in the electric arc; it is here very distinct, even with a small condenser. The existence of 413.2 was foretold by M. Lecoq de Boisbaudran, who calculated its existence from theoretical considerations, before discovering it in a concentrated solution; it is much easier to detect in melted Li_2CO_3 . In the condenser spark it is, on the other hand, very wide and cloudy.

The principal papers relating to the spectrum of potassium are based on experimental conditions very different to those under which I have worked; that is to say, the action of a strong condensation effecting the complete dissociation of a melted salt. By this means I obtained, united in one spectrum, the different lines which have been detected singly, by very different methods, and between which we can notice the relations between relative intensities; these do not resemble the corresponding lines found in the preceding researches.

M. Lecoq de Boisbaudran worked with a non-condensed spark on fused sulphate of potassium; Sir William Huggins and MM. Eder and Valenta with slight condensation at the ordinary temperature; Profs. Liveing and Dewar and MM. Kayser and Runge, in the electric arc, where the spectrum of potassium is notably less rich in lines than in the spark. The electrical and optical arrangements here used were the same as those in my previous experiments already described.

Potassium.

δ	769.9	Difficult to see.
	766.6	
γ	693.9	Fairly strong.
	691.4	Well marked.
	630.8*	" "
	624.55*	Easily seen.
	611.75†	Very well marked.
	583.2	Strong.
α	581.1	Fairly well seen.
	580.1	Strong.
	578.3	Very well marked.
	551.5*	Weak.
	536.0	Easily seen, diffuse.
β	534.4	Fairly well seen, diffuse.
	534.0	Easily seen, diffuse.
	532.35	Fairly well seen, diffuse.
	511.3	Fairly well seen, diffuse, and almost confounded with one another.
η	509.9	
	505.1‡	Weak.
	500.7	Weak.
	485.55§	Fairly well seen.
	485.1§	" "
ζ	482.9	Fairly strong.
	465.2	Fairly well seen.
	460.6¶	" "
	450.65¶	Weak.
	446.6	" "
	438.8¶	Fairly well seen.
	430.95	" "
	430.65¶	" "
	426.4§	Fairly strong.
	422.55	Fairly well seen.
	422.25	" "
	421.0	Weak.
	418.55¶	Fairly strong, wide.
ϵ	404.55	Strong, very wide, diffuse.

* Seen by Sir William Huggins only.

† Seen by Sir William Huggins, afterwards by M. Lecoq de Boisbaudran only.

‡ Seen by M. Lecoq de Boisbaudran only.

§ Seen by Profs. Liveing and Dewar only.

|| Seen by MM. Eder and Valenta only.

¶ Seen by M. Lecoq de Boisbaudran, afterwards by MM. Eder and Valenta only.

Several lines in the violet, detected by MM. Eder and Valenta by means of photography, which is more sensitive than the eye in this part of the spectrum, were not visible to me.

To get greater accuracy with regard to wave-lengths, we can refer to the memoir by MM. Kayser and Runge on the arc spectrum of potassium (*Abhand. Berliner d. Akad.*, 1890), and for the lines which do not show in the arc, to the work of MM. Eder and Valenta already quoted. All these observers had at their disposition either a Rowland's apparatus or a spectrograph—instruments which are not to be found at the Faculté des Sciences in Paris. The approximation here given, however, amply suffices for the identification of the lines in any current research in a chemical laboratory.

The classic red lines, K δ , situated at the end of the spectrum, always rather diffused, are only visible when working with instruments of little absorption; further, they are not mentioned in the work of Sir William Huggins or of M. Thalén. The double line, K γ , on the contrary, is very bright and characteristic; the second, or more narrow line, is slightly fainter than its companion. There are, again, three lines in the red—630.8, 624.55, and 611.75—which, though mentioned, but considered to be doubtful by Sir William Huggins, were either not seen by subsequent observers, or else attributed by them to impurities. However, the strongest and most refrangible, 611.75, was seen, though very faintly, by M. Lecoq de Boisbaudran, using a simple spark only, with pure fused sulphate of potash. MM. Eder and Valenta con-

sidered these rays as not belonging to potassium, but made no endeavour to define their origin. I have always found them in company with the double line $K\gamma$ in melted salts of potassium, when using a sufficient condensation. I repeat, that I have always succeeded in obtaining them eventually with complete absence of lines of other foreign matters; I consider them, therefore, as characteristic of the spectrum of dissociation of potassium in its salts.

The brightest group is $K\alpha$, in the green, and appears to show the most sensitive reaction of potassium in the spark. $K\beta$ is fainter and diffuse, and the two middle lines cannot be separated with a single prism. $K\eta$ is still fainter, and more diffuse than under ordinary conditions; it is easily seen as a single band about $510\cdot5$.

In the blue, K is, on the contrary, sensibly stronger and becomes characteristic: this line is, however, entirely missing in the arc.

In the same manner, $426\cdot4$ in the indigo, and $418\cdot55$ in the violet, become more intense with condensation, the augmentation of which makes itself particularly felt in this region of the spectrum. $K\epsilon$, the last line visible, is a double line which, in the condenser spark, becomes transformed into a strong diffuse band which cannot be divided; it is very bright and characteristic of potassium. — *Bull. Soc. Chim.*, Series 3, vols. xvii.-xviii., Nos. 16-17.

THE COMBUSTION OF ORGANIC SUBSTANCES IN THE WET WAY.*

By I. K. PHELPS.

In a former paper (*Am. Journ. Sci.*, vol. ii., p. 70) I have shown that carbon dioxide may be estimated iodometrically with a fair degree of accuracy. Inasmuch as this method is not dependent upon the rate of flow or rapidity of generation of the carbon dioxide, it seemed possible that some advantage might follow its application to the determination of organic carbon, oxidised by liquid reagents.

Method of Oxidation by Potassium Permanganate.

The first experimental test in this direction was made with oxalic acid, which was oxidised according to the well-known reaction of potassium permanganate in the presence of sulphuric acid. The apparatus used was the same as that previously described in the iodometric process referred to above. It consisted, in the main, of an evolution flask and an absorption flask, properly connected. As an evolution flask, a wide-mouthed flask of about 75 c.m.³ capacity was used. This was closed by a doubly perforated rubber stopper, carrying a separating funnel for the introduction of liquid into the flask and a glass tube of 0·7 c.m. internal diameter, which was expanded to a small bulb just above the stopper, to carry off the gas. This exit tube was joined by means of a rubber connector to a tube which passed through the rubber stopper of the absorption flask, which was an ordinary round-bottomed flask of 250 c.m.³ capacity. This tube ended in a valve of the Kreider pattern (*Am. Journ. Sci.*, l., p. 132), which was enclosed in a larger tube, reaching nearly to the bottom of the absorption flask. The second hole of the stopper of this absorption flask was filled by a glass tube closed by a rubber connector and screw pinch-cock.

The barium hydroxide solution for use in the determination of the carbon dioxide was prepared by filtering a cold saturated solution of the commercial salt into a large bottle, which was connected with a self-feeding burette. The solution was standardised in the manner described in my former paper, by boiling with an excess of decinormal iodine solution in an ether wash-bottle. The short tube of the glass ground stopper of the bottle

was sealed to a Will and Varrentrapp absorption apparatus, which was charged during the operation with a solution of potassium iodide to prevent the loss of elementary iodine in the boiling; the long tube of the bottle was used as an inlet tube, and was closed externally by a rubber cap during the boiling. After cooling, the excess of iodine used was determined by titration with decinormal arsenious acid solution and the iodine lost calculated on barium hydroxide molecule for molecule.

Potassium permanganate was prepared for use by dissolving the commercial salt in water, and boiling this solution made acid with sulphuric acid, until free from carbon dioxide. Water was also prepared free from carbon dioxide by boiling distilled water until one-third had been driven off in steam and was kept until used in full-stoppered flasks.

For the first determinations of carbon, crystallised ammonium oxalate was weighed out and introduced into the boiling flask with 10 to 15 c.m.³ of pure water and the flasks connected as described above with an appropriate amount of barium hydroxide solution (3 to 5 c.m.³ in excess of the amount required to precipitate the carbon dioxide to be determined) in the absorption flask. The whole system was then evacuated with the water-pump to a pressure of 200–225 m.m. and the oxalate solution in the boiling-flask warmed. An excess of potassium permanganate solution was then run in through the funnel tube and the mixture warmed again, when the oxidation of the oxalate was shown by the carbon dioxide evolved. The carbon dioxide was completely set free by the introduction of 10 c.m.³ of sulphuric acid (1 : 4) and was driven completely to the absorption flask by boiling for five minutes. During the passage of the gas into the absorption-flask, it was shaken frequently and was kept cool by standing in a dish of water and by pouring cold water over it from time to time. If, during the boiling, any fears are entertained as to the strength of the vacuum in the flasks, they may be easily allayed by opening momentarily the stopcock of the funnel-tube and noting the direction of the flow of water contained in the funnel. After the boiling was ended, the atmospheric pressure was restored by allowing air, purified from carbon dioxide by passage through potash bulbs, to enter through the funnel-tube of the boiling flask. Then the flasks were disconnected and the stopper of the absorption-flask with its attachments was removed, the valve and its tube being carefully washed free from barium hydroxide. A second stopper, which was provided with a separating funnel, and a Will and Varrentrapp absorption apparatus, containing water to serve as a trap, was inserted into the mouth of the absorption flask and the emulsion brought to the boiling point. Decinormal iodine solution was then run in through the funnel-tube in sufficient quantity to destroy the larger part of the excess of barium hydroxide, and the emulsion brought to the boiling-point again, after which iodine was again run in, but this time to the permanent red colour of the excess of free iodine. After cooling, this excess of iodine was determined by titration with decinormal arsenious acid solution. Thus, the excess of barium hydroxide used being determined by the iodine lost, the barium hydroxide used, now in the form of carbonate, was known, from which the carbon dioxide which precipitated this carbonate may be calculated.

The following results were obtained by this procedure.

TABLE I.

	Ammonium oxalate taken. Grm.	BaO ₂ H ₂ taken. Grm.	BaO ₂ H ₂ found. Grm.	CO ₂ found. Grm.	CO ₂ calculated. Grm.	Error on CO ₂ . Grm.
1.	0·2522	0·7267	0·1170	0·1565	0·1561	0·0004+
2.	0·2542	0·7267	0·1113	0·1579	0·1574	0·0005+
3.	0·5020	1·4535	0·2417	0·3110	0·3108	0·0002+
4.	0·5058	1·3954	0·1753	0·3131	0·3131	0·0000±
5.	1·0033	2·6163	0·1955	0·6213	0·6211	0·0002+
6.	1·0003	2·5951	0·1836	0·6189	0·6192	0·0003-
7.	1·0010	2·6163	0·2037	0·6192	0·6197	0·0005-

* Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, Series 4, Vol. iv., No. 23, November, 1897.

In Experiments 5 and 6 a few drops of ammonia were added to the oxalate solution before running in the permanganate; in 3 and 7, the permanganate was treated to alkalinity with barium hydroxide; in the remaining experiments, 1, 2, and 4, the permanganate was slightly acid with the sulphuric acid used in its purification from carbon dioxide, as already described. The results obtained are good, and it is plain that the oxidation proceeded regularly, whether the first action of the permanganate was in the alkaline or slightly acid solution.

Jones (*Am. Chem. Journ.*, xvii., 539) has shown that formates may be determined volumetrically by titration with potassium permanganate in alkaline solution. In an attempt to determine formates by the process outlined above, the pure barium salt was used. This was prepared by treating the aqueous solution of formic acid with pure barium carbonate to neutrality and crystallising the product. It was proven pure by ignition and weighing in the form of carbonate.

In making determinations of carbon in this formate, weighed portions were introduced into the boiling flask, together with sodium hydroxide solution, which was taken in such quantity as to more than neutralise the acid in the potassium permanganate. Naturally, the sodium hydroxide must be free from carbonate—which was effected by treatment with an excess of barium hydroxide and filtering. An excess of potassium permanganate is then run into the flask and the solution heated to boiling. An excess of dilute sulphuric acid is introduced into the mixture, and the carbon dioxide thus set free completely driven over to the absorption flask and determined as before. Table II. shows results obtained by the process.

TABLE II.

	Barium formate taken. Grm.	BaO ₂ H ₂ taken. Grms.	BaO ₂ H ₂ found. Grm.	CO ₂ found. Grm.	CO ₂ calculated. Grm.	Error on CO ₂ . Grm.
1.	0.5001	0.9302	0.1745	0.1939	0.1935	0.0004+
2.	0.5033	0.9112	0.1402	0.1953	0.1947	0.0006+
3.	1.0002	1.6861	0.1793	0.3867	0.3870	0.0003-
4.	1.0059	1.6279	0.1093	0.3897	0.3892	0.0005+
5.	1.3750	2.2529	0.1820	0.5315	0.5320	0.0005-
6.	1.5028	2.4419	0.1754	0.5816	0.5814	0.0002+

These results show plainly that the carbon of formic acid may be determined accurately by the method outlined.

It was found incidentally that ammonia cannot take the place of the sodium hydroxide in this process, probably because the ammonia volatilises to the absorption flask during the boiling and is acted on by the iodine subsequently used, and is thus registered as barium hydroxide.

It is a well known fact that tartrates are oxidised by permanganates. I have found, however, that when tartaric acid is treated under the conditions of analysis outlined above in acid solution, the oxidation is incomplete; but that oxidation is complete if the tartrate is heated in a solution alkaline with sodium hydroxide and then acidified with sulphuric acid.

The tartrate used was a re-crystallised tartar emetic, dried at 100° C. The following results were obtained with such a tartrate by this process.

TABLE III.

	Tartar emic taken. Grm.	BaO ₂ H ₂ taken. Grms.	BaO ₂ H ₂ found. Grm.	CO ₂ found. Grm.	CO ₂ calculated. Grm.	Error on CO ₂ . Grm.
1.	0.5051	1.2450	0.1709	0.2756	0.2751	0.0005+
2.	0.5030	1.2226	0.1536	0.2743	0.2739	0.0004+
3.	0.7509	1.7355	0.1401	0.4094	0.4091	0.0003+
4.	0.7541	1.7430	0.1410	0.4111	0.4107	0.0004+
5.	1.0018	2.3456	0.2187	0.5458	0.5456	0.0002+
6.	1.0005	2.2435	0.1196	0.5451	0.5450	0.0001+

It seems possible to draw the general conclusion from the results recorded that organic substances which are

oxidised completely by the permanganate may be determined by the process outlined above. It will also be seen that the use of the rubber stopper in the boiling flask, with due care to prevent its contact with the solution, does not introduce an appreciable error.

Wanklyn and Cooper (*Phil. Mag.*, [5], vii., 138) and others have noted the fact that potassium permanganate, whether in acid or alkaline solution, will not oxidise all organic substances (acetates, for example), even at the boiling temperature. It is well known that a mixture of concentrated sulphuric and chromic acids has a much wider field of action in oxidising organic compounds than the permanganate. With hopes of applying this reagent more widely to the determination of organic carbon, the experiments about to be recorded were tried.

(To be continued).

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING OCTOBER 31ST, 1897.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolitan Water Act, 1871.

London, November 10th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 182 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from October 1st to October 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 182 samples examined during the month all were found to be clear, bright, and well filtered.

The rainfall at Oxford during the month shows a great deficiency, only 1.22 inches of rain having fallen, while the average for the last 30 years is 2.75 inches, making a deficit of 1.53 inches. The total rainfall for the year now shows a deficit of 0.41 inch.

Our bacteriological examination of 253 samples taken by us have given the following results; we have also examined 72 other samples, from stand-pipes, special points, wells, &c., making a total of 325 samples in all:—

	Microbes per c.c.
Thames water, unfiltered (mean of 26 samples)	3547
Thames water, from the clear water wells of five Thames-derived supplies (mean of 124 samples)	51
Ditto ditto highest	434
Ditto ditto lowest	0
New River, unfiltered (mean of 26 samples)	397
New River, filtered (mean of 26 samples)	35
River Lea, unfiltered (mean of 26 samples)	1588
River Lea, from the clear water well of the East London Water Company (mean of 25 samples)	39

The filtration and settling appliances of all the Companies have been in a high state of efficiency during the past month.

The water of every Company has been specially tested for pathogenic organisms, but with negative results.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.

JAMES DEWAR.

ELECTROLYTIC SEPARATION OF NICKEL AND COBALT FROM IRON. APPLICATION TO THE DETERMINATION OF NICKEL IN STEEL.

By O. DURN.

THE accurate separation of nickel, and nickel and cobalt, from large quantities of iron presents notable difficulties. The great number of methods already published proves that no really satisfactory solution of the difficulty has been reached.

For some years nickel steels have taken an important industrial rank, whence an expeditious and precise method presents a certain interest. In his chemical study on the methods of analysis of irons and cast metals, Ad. Carnot gives the preference to the method of Rothe, and this method, which has been simultaneously devised in France by Hanrich, depends on the separation of ferric chloride in an acid solution by means of ether. Latterly Pinerua has modified this method. He uses, at a low temperature, ether saturated with hydrochloric acid.

It is easy to arrive at the same result by means of electrolysis. If we precipitate by ammonia in excess a ferric solution containing, e.g., nickel, a part of this metal is carried down by the ferric hydrate. If we submit to electrolysis the ammoniacal liquid holding the precipitate in solution, we may obtain on the kathode the entire deposit of the nickel. The separation is not absolutely accurate. Almost always a minute quantity of iron is also deposited upon the kathode, but in suitable conditions this quantity ranges about 1 to 2 m.grms. when the iron present may reach 400 to 500 m.grms. For precise experiments it is necessary to make correction in the weight of the metal deposited, which is easily done by dissolving the deposit in hydrochloric acid and precipitation with ammonia after for oxidising.

The use of the nitric solution, which under the like conditions enabled M. Riche to separate copper from iron, presents certain inconveniences. It is the same with the hydrochloric solution. We obtain good results by operating on the sulphuric solution to which ammonium sulphate has been added. The following is the process:—The solution containing nickel and iron as per-salts, with the addition, if needful, of a slight excess of sulphuric acid, is evaporated to dryness, is then taken up in a minimum of water with the addition of 5 to 10 grms. ammonium sulphate, and heated until a clear solution is obtained. This liquid is poured whilst stirred into the crucible of Riche's apparatus, in which have been placed 60 to 70 c.c. of concentrated ammonia.

As a source of electricity we use two or three accumulators, arranged in tension so as to regulate the intensity of the current at the outset at from 1.5 to 2.5 ampères. Under these conditions the nickel is entirely deposited in less than four hours.

The method has been checked by means of standardised solutions of iron and nickel, some of the results being—

	Iron.	Ni (added).	Metal deposited.	Correction for iron.	Ni recovered.	Difference.
1.	404.3	29.8	30.8	1.0	29.8	0.0
2.	269.5	74.6	75.9	1.3	74.6	0.0
3.	269.5	149.2	150.1	0.8	149.3	+0.1

The same procedure is equally applicable to cobalt:—

4.	404.2	62.5	63.4	1.9	61.5	-1.0
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Determination of Nickel in Steels.—We attack from 250 to 300 m.grms. of the sample with aqua regia in a porcelain capsule. When the action is complete we add 1 c.c. of sulphuric acid, and evaporate until white fumes are produced.—*Comptes Rendus*, cxxv., No. 11.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

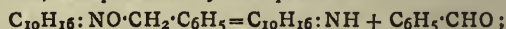
THE following are the abstracts of papers received during the vacation, and published in the *Transactions*:—

96. "The Ethers of Camphoroxime." By M. O. FORSTER, Ph.D.

The methyl ether of camphoroxime boils at 181.5°–182.5° under a pressure of 357 m.m.; it has the sp. gr. = 0.9631, and the specific rotatory power $[\alpha]_D = -13.05^\circ$ at 20°. It does not reduce an ammoniacal solution of silver nitrate, and dissolves in mineral acids without undergoing change. The nitrate crystallises from benzene in needles melting at 81°–82°, and has $[\alpha]_D = -16.9^\circ$ in benzene; the hydriodide is amorphous, and melts at 157° with vigorous effervescence.

The ethyl ether (Nigeli) boils at 185° under a pressure of 336 m.m., and has the sp. gr. = 0.9470; the specific rotatory power $[\alpha]_D = -19.0^\circ$ at 23.5°.

The benzyl ether is a colourless oil, which on distillation is in part resolved into benzaldehyde and camphorimine, as represented by the equation—



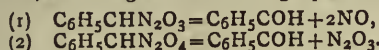
it has $[\alpha]_D = -16.4^\circ$ in alcohol, and forms an amorphous hydriodide which melts at 91°. Concentrated sulphuric acid resolves the ether into camphoroxime and the resinous hydrocarbon, $C_{14}H_{12}$, obtained on dissolving benzylic alcohol in concentrated sulphuric acid. Alcoholic hydrochloric acid eliminates α -benzylhydroxylamine from the ether; the platinochloride of this base forms golden-yellow scales, and does not melt below 250°.

The acetyl derivative of camphoroxime is a colourless liquid, and is completely converted into acetic acid and campholenitrile on distillation; it has the specific rotatory power $[\alpha]_D = -45.8^\circ$ in alcohol, and on treatment with cold phenylhydrazine yields symmetrical acetylphenylhydrazine and camphoroxime.

The benzoyl derivative crystallises from acetone in magnificent six-sided prisms, and melts at 88°–90°; it has $[\alpha]_D = -40.7^\circ$ in alcohol, and yields benzanilide when heated with aniline. Cold phenylhydrazine gives rise to benzoylphenylhydrazine and camphoroxime. Camphoroxime hydrobromide melts and evolves hydrogen bromide at 174°; it has $[\alpha]_D = -35.8^\circ$ in alcohol, and is converted by glacial acetic acid into campholenitrile and hydrogen bromide. Camphoroxime platinochloride crystallises in transparent prisms which become opaque in the desiccator, and melts at 156.5° with vigorous effervescence; cold water regenerates the oxime. Inactive camphoroxime melts like the active modification at 118°; it crystallises from petroleum in diamond-shaped plates, and is racemic according to the classification recently suggested by Kipping and Pope (*Proc. Chem. Soc.*, 1897, p. 135).

97. "The Action of Nitrogen Trioxide and Tetroxide on Alcohols." Part I. By JULIUS BEREND COHEN, Ph.D., and HARRY THORNTON CALVERT, B.Sc.

The authors have found that when nitrogen trioxide or tetroxide dissolved in chloroform is allowed to act upon benzyl alcohol, that water is in both cases eliminated, and compounds of the formula $C_6H_5CHN_2O_3$ and $C_6H_5CHN_2O_4$ are probably formed, which rapidly decompose on standing into benzaldehyde, with the separation in the first case of nitric oxide, and in the second of nitrogen trioxide, according to the following equations:—



The latter substance, which may be termed benzylidene nitrosate, is decomposed by water into a compound of the formula $C_7H_7NO_3$, which is probably identical with a substance obtained by Lippmann and Hawliczek (*Ber.*, 1876, ix., 1463) by the action of nitric acid upon benzaldehyde. By the action of reducing agents it is converted into benzyl alcohol, benzylamine, and ammonia.

98. "The Action of Nitrogen Tetroxide on Ortho- and Para-nitrobenzylalcohol." By JULIUS B. COHEN, Ph.D., and WILLIAM H. HARRISON, B.Sc.

The authors have discovered a simple method for preparing the aldehydes corresponding to ortho- and para-nitrobenzylalcohol. The reaction consists in treating the alcohol with a small quantity of nitrogen tetroxide in presence of air. A nearly theoretical yield of these aldehydes, hitherto very difficult to prepare, has been effected by this method.

99. "The Action of Aromatic Amines upon Diacetyl-tartaric Anhydride." By JULIUS BEREND COHEN, Ph.D., and WILLIAM HUDSON HARRISON, B.Sc.

In attempting to prepare the isomeric toluidio-acetyl tartaric acids, by acting upon diacetyl tartaric anhydride with the isomeric toluidines, with a view to comparing their optical characters, the authors were unsuccessful; but obtained, on the other hand, by this reaction with different aromatic amines, a series of golden-yellow crystalline compounds. The formula of the aniline compound is probably $C_{16}H_{12}N_2O_3$, that of the paratoluidine compound $C_{18}H_{16}N_2O_3$, and of the α -naphthylamine compound $C_{27}H_{16}N_2O_3$. The constitution of these compounds has not yet been ascertained. The reaction in all cases is very complex, and the yield of the yellow substances very small.

100. "Studies on Citrazinic Acid." Part V. By W. J. SELL, M.A., and F. W. DOOTSON, B.A.

This investigation was commenced with the view of obtaining some evidence of the positions of the hydroxyl groups in citrazinic acid, by preparing the corresponding dichlorisonicotinic acid, and then replacing the chlorine atoms either by cyanogen or methyl, and thus by well-known methods obtaining one of the tricarboxy-acids whose constitution has been established. In the preparation of the dichlorisonicotinic acid by the interaction of phosphorus pentachloride on citrazinic acid, however, such a number of interesting substances were found to be produced that it was determined to publish this part of the work at once, leaving the remainder for a further communication. The following substances, amongst others, have been isolated, and are described in the paper:—

(1) Chlorhydroxyisonicotinic acid; (2) dichlorisonicotinic acid; (3) tetrachlorisonicotinic acid chloride; (4) $\alpha\beta\alpha'\beta'$ -tetrachlorisonicotinic acid; (5) tetrachlorpyridine; (6) pentachlorpyridine; (7) pentachlorpicoline.

101. "The Condensation of Chloral with Resorcinol." Part II. By J. T. HEWITT, M.A., D.Sc., and FRANK G. POPE.

In an earlier paper (*Trans.*, 1896, lxix., 1265) the view was expressed that the substance of the formula $C_{14}H_{10}O_5$, obtained by the condensation of chloral

hydrate with resorcinol, was a lactone of 2:4:2':4'-tetrahydroxydiphenylacetic acid. The authors had overlooked a paper by Michael and Comey (*Amer. Chem. Journ.*, 1883-4, v., 350), in which the formula $C_8H_6O_3$ was attributed to the compound in question. The determination of the molecular weight by the lowering of freezing-point of a phenolic solution gave as result 232; the values required by the formulæ $C_8H_6O_3$ and $C_{14}H_{10}O_5$ being 150 and 258 respectively. The analyses of the acetate and benzoate have further confirmed the authors' views that the compound possesses three hydroxyl groups. In addition to this, it has been found that a red salt is precipitated when an excess of sodium ethylate solution is added to an absolute alcoholic solution of the lactone: the salt was found to contain 22.10 per cent of sodium, whilst the formula $C_{14}H_{10}O_5Na_3$ requires 21.30 per cent of sodium.

The analysis of the salt, obtained by boiling the lactone with water and barium carbonate, led to the formula $Ba(C_{14}H_{11}O_6)_2$; the soluble zinc salt obtained in a similar way gave a percentage of zinc which agrees with the formula $Zn(C_{14}H_{11}O_6)_2$.

102. "On β -oxycellulose." By BENJAMIN SAMUEL BULL, M.A., B.Sc.

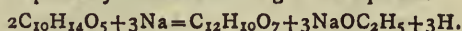
β -oxycellulose has been studied by several workers, and Cross and Bevan have prepared a trinitro-derivative. In this paper a benzoate and nitrate obtained from β -oxycellulose are described. These compounds are probably hexa-derivatives of a substance having the empirical formula $C_{16}H_{22}O_{14}$.

103. "A New Synthesis of Phloroglucinol." By DAVID S. JERDAN, B.Sc.

When finely divided sodium is dissolved in a benzene solution of ethylic acetone-di-carboxylate, and the solution is boiled for some hours, a gummy deposit is slowly formed. The whole is then shaken with water, and the aqueous solution, after separation of the benzene, is acidified with sulphuric acid. The solution becomes milky, and a granular precipitate falls after a short time. The new substance may be re-crystallised from glacial acetic acid, and then possesses a composition corresponding with the formula $C_{12}H_{10}O_7$.

This compound, when boiled with methylic alcohol containing 3 per cent of hydrogen chloride, took up a molecule of alcohol, giving a crystalline ester, $C_{13}H_{14}O_8$. The substance $C_{12}H_{10}O_7$ must therefore be a lactone. Further, on hydrolysis with baryta solution, the lactone gave carbon dioxide, alcohol, malonic acid, and phloroglucinol. The new compound is therefore a phloroglucinol derivative.

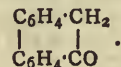
It is probably formed according to the equation—



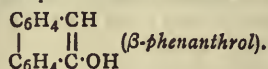
The immediate product of the reaction must of course be a sodium derivative, the lactone $C_{12}H_{10}O_7$ being formed from this on addition of sulphuric acid.

104. "Phenanthrone." By FRANCIS R. JAPP, F.R.S., and ALEXANDER FINDLAY, M.A., D.Sc.

Phenanthrone was regarded by its discoverer, Lachowicz (*J. Pr. Chem.*, 1883, [2], lxviii., 173), as a ketone of the formula—



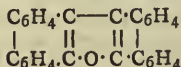
Japp and Klingemann (*Trans.*, 1893, lxiii., 770) suggested that it might be a phenol of the formula—



In the hope of deciding between these two formulæ, the present authors have prepared various derivatives of phenanthrone. The evidence, however, points in both directions. The compound reacts both in the ketonic

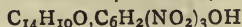
and in the phenolic form, although more frequently in the latter. In the great majority of its reactions it is a strict analogue of β -naphthol.

In preparing phenanthrone by the method discovered by Japp and Klingemann—reduction of phenanthraquinone with hydriodic acid—the authors find that two other substances are simultaneously formed: β -phenanthrylic oxide, $(C_{14}H_9)_2O$ (m. p. 210°), and tetraphenylene-furfuran,—

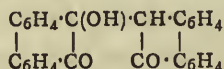


(m. p. 306°). The latter was obtained by Japp and Klingemann by the destructive distillation of monacetyl phenanthraquinol.

Phenanthrone and β -phenanthrylic oxide both yield molecular compounds with picric acid,—



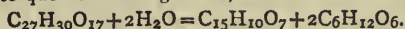
(m. p. 185°) and $(C_{14}H_9)_2O, 2C_6H_2(NO_2)_3OH$ (m. p. 148°). Phenanthrone, when in solution, is converted by aerial oxidation into the compound $C_{28}H_{18}O_3$ (obtained by another process by Japp and Klingemann), which crystallises in dark red laminae melting at $156-157^\circ$. This compound is broken up by acetic anhydride into phenanthrone and phenanthraquinone, the former undergoing acetylation. It may be synthesised by the direct union of phenanthrone and phenanthraquinone. The authors regard it as an aldol condensation compound of these two substances, and ascribe to it the constitution—



On boiling with fuming hydriodic acid, it is converted quantitatively into tetraphenylene-furfuran. Acetic anhydride converts phenanthrone into β -phenanthrylic acetate, $C_{14}H_9 \cdot O \cdot C_2H_3O$ (m. p. $77-78^\circ$). When heated with methylic alcohol and sulphuric acid, phenanthrone yields methylic- β -phenanthrylic oxide, $C_{14}H_9 \cdot O \cdot CH_3$ (m. p. $96-97^\circ$). When heated with ammoniac it yields a mixture of β -phenanthryllamine, $C_{14}H_9 \cdot NH_2$ (m. p. 139°), and β -diphenanthryllamine, $(C_{14}H_9)_2NH$ (m. p. 237°). With phenylhydrazine at 200° it interacts, eliminating water and ammonia and yielding 2':3'-diphenyleneindole (m. p. $188-189^\circ$).

105. "The Yellow Colouring Principles of various Tannin Matters." IV. By A. G. PERKIN.

Cape sumach, the leaves of the *Colpoen compressum*, is used in South Africa as a substitute for sumach (*Rhus Coriaria*) under the name of "Pruim-bast." According to H. Procter (private communication), it contains 23 per cent of a catechol tannin. Its dyeing property is due to the presence of a new glucoside, *osyritrin*, $C_{27}H_{30}O_{17}$, pale yellow needles, m. p. 185° , which is decomposed by acid into quercetin and glucose,—



This is not identical with viola-quercetrin (Mandelin, *Z.*, 1883, 1369), $C_{42}H_{42}O_{24}$, which exists in the *Viola tricolor florensensis*. The tannin, obtained as an orange-coloured transparent mass, is a glucoside yielding, with acid, an anhydride or phlobophane and a sugar. By fusion with alkali, protocatechuic acid is formed. A re-examination of gambier catechu (*Ungarica Gambier*) corroborated the statement of Löwe (*Zeit. Anal. Chem.*, 1874, xii., 127) that this contains quercetin, *Acacia catechu* not previously examined was found to contain the same colouring matter.

The dyeing properties of a commercial sample of Venetian sumach (*R. Cotinus*) are due to myricetin and not to quercetin as stated by Löwe (*loc. cit.*). This result will be corroborated by the examination of a specially picked sample.

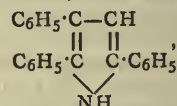
Valonia (*Quercus Ægilops*), divi-divi (*Casalpina*

Coriaria), myrabolans (*Terminalia chebula*), agarobilla (*Casalpina brevifolia*), pomegranate rind (*Punica granatum*), and gall-nuts (*Quercus infectoria*), owe their tinctorial property to ellagic acid, and contain no member of the quercetin group. It is here pointed out that the plants examined hitherto contain, respectively, a tannin and colouring matter which yield on decomposition identical acids, and in some cases the same phenol.

106. "Ammonia and Phenylhydrazin Derivatives of $\alpha\beta$ -Dibenzoylcinnamene (Anhydracetophenonebenzil)." By FRANCIS R. JAPP, F.R.S., and ALFRED TINGLE, B.Sc.

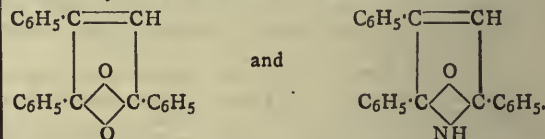
By oxidising dibenzoylcinnamenimide, $C_{22}H_{17}NO$ —the first product of the action of ammonia on dibenzoylcinnamene—with chromium trioxide, the authors have obtained a mixture of dibenzamide, benzamide, and regenerated dibenzoylcinnamene.

By reducing dibenzoylcinnamenimide with zinc dust and acetic acid in the cold, A. Smith's triphenylpyrrhole,—



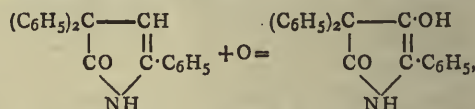
melting at $140-141^\circ$ (*Trans.*, 1890, lvii., 645), is formed.

The authors discuss the various reactions of dibenzoylcinnamene and dibenzoylcinnamenimide, and ascribe to these compounds the formulæ—



It seems to be impossible to assign to dibenzoylcinnamenimide, for example, any other formula which will account for the formation of dibenzamide during oxidation.

By the oxidation of triphenylpyrrholone—the transformation product of dibenzoylcinnamenimide under the influence of heat—with chromium trioxide, the authors have obtained a compound which they regard as triphenylhydroxy-pyrrholone (m. p. 168°),—



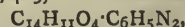
or one of its possible tautomeric forms. Heated with caustic potash, this compound evolves ammonia, and yields a mixture of benzoic and benzoic acids.

The authors have also studied the destructive distillation of the compound $C_{28}H_{22}N_2$ (m. p. about 230°), obtained by Japp and Huntly (*Trans.*, 1888, liii., 184) by the action of phenylhydrazin on dibenzoylcinnamene. They find that it yields the 1:3:4-triphenylpyrazole obtained by A. Smith (*Annalen*, 1896, cclxxxix., 332) by the destructive distillation of tetraphenyldihydro-1:2-diazine. They point out that result renders it very improbable that the compound $C_{28}H_{22}N_2$ has the constitution of an anilidotriphenylpyrrhole, ascribed to it by Japp and Klingemann (*Trans.*, 1890, lvii., 671).

107. "Derivatives of Cotoin and Phloretin." By A. G. PERKIN and H. W. MARTIN.

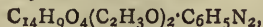
A study of the acetylation of the diazobenzene derivatives of cotoin and phloretin.

Cotoin, $C_{14}H_{12}O_4$, a constituent of coto-bark, is, according to Ciamician and Silber, a monomethyl ether of benzoylphloroglucinol, $C_6H_2(OCH_3)(OH)_2 \cdot CO \cdot C_6H_5$ (*Ber.*, 1894, xxvii., 409). Cotoinazobenzene,—



forms orange-yellow needles, m. p. $183-184^\circ$. Cotoin-azo-o-toluene, $C_{14}H_{11}O_4 \cdot CH_3 \cdot C_6H_4N_2$, m. p. $203-204^\circ$,

and *cotoin-azo-*p*-toluene*, m. p. 207—208°, crystallise similarly. *Diacetyl-azo-benzene cotoin*,—



crystallises in scarlet needles, m. p. 155—156°. As with the maclurin compound (*Trans.*, 1897, lxxi., 186), the acetyl-groups could be determined by Liebermann's method.

Phloretin, $\text{C}_{15}\text{H}_{14}\text{O}_5$, occurs in the root bark of the apple tree as a glucoside phloridzin. According to Ciamician and Silber, it has the constitution $\text{C}_6\text{H}_2(\text{OH})_3\cdot\text{CO}\cdot\text{CH}(\text{CH}_3)\cdot\text{C}_6\text{H}_4\cdot\text{OH}$. *Phloretin-disazobenzene*, $\text{C}_{15}\text{H}_{12}\text{O}_5(\text{C}_6\text{H}_5\text{N}_2)_2$, red needles, m. p. 254—256°, *phloretin-disazo-*o*-toluene*, m. p. 250—251°, and *phloretindisazo-*p*-toluene*, m. p. 250—251°, closely resemble the corresponding maclurin derivatives. *Acetyl phloretindisazobenzene*, $\text{C}_{15}\text{H}_{11}\text{O}_5(\text{C}_2\text{H}_3\text{O})(\text{C}_6\text{H}_5\text{N}_2)$, forms orange-red needles melting at 217—219°. No higher acetyl derivative could be obtained. Comparing this result with those previously obtained with maclurin and phloroglucinoldisazobenzenes (*Trans.*, 1897, lxxi., 186), it would thus appear that phloretin contains only three hydroxyl groups. From Ciamician and Silber's work there appears to be no doubt, however, as to the correctness of their constitution for phloretin (*loc. cit.*). Thus, all hydroxyls in the phloroglucinol nucleus of phloretin must in diazobenzenephloretin be in the ketonic form, a peculiarity which in some way is therefore due to the influence of the phloretol group.

108. "*Azobenzene Derivatives of Phloroglucinol*." By A. G. PERKIN.

Though phloroglucinol is known to yield azo- and disazo-derivatives as phloroglucinol-*p*-azobenzene sulphonic acid, $\text{C}_6\text{H}_5\text{O}_3\cdot\text{N}_2\cdot\text{C}_6\text{H}_4\cdot\text{SO}_3\text{H}$ (Stebbins, *Am. Chem. Soc. J.*, 1880, ii., 240), and disazobenzenephloroglucinol, $\text{C}_6\text{H}_4\text{O}_3(\text{N}_2\cdot\text{C}_6\text{H}_5)_2$ (Weselsky and Benedikt, *Ber.*, 1879, xii., 226), no trisazo-compounds have been previously obtained, though judging from its constitution the formation of such should be expected.

Phloroglucinoltrisazobenzene, $\text{C}_6\text{H}_5\text{O}_3(\text{C}_6\text{H}_5\text{N}_2)_3$, fine needles possessing a green iridescence which do not melt below 300°, is formed by addition of diazobenzene sulphate to a solution of phloroglucinol in aqueous sodium carbonate. Its production is independent of the amount of diazobenzene sulphate employed. It contains no free hydroxyl groups, being insoluble in alkaline solutions.

*Phloroglucinol-*o*-trisazoanisol* is prepared from phloroglucinol and *o*-diazooanisol in either sodium carbonate or acetate solution. No corresponding disazo-compound could be obtained in this manner. It forms maroon coloured needles melting above 300°, insoluble in alkaline solutions.

*Phloroglucinol-disazobenzene-*m*-azonitrobenzene*, obtained from phloroglucinol-disazobenzene and *m*-diazonitrobenzene, forms dull red needles, m. p. 290°.

It is proposed to study the reaction of other substituted diazobenzenes with phloroglucinol under similar conditions.

109. "*The Action of Phosphorus Pentachloride on Fenchone*." By J. ADDYMAN GARDNER, M.A., and G. B. COCKBURN, B.A.

Fenchone is acted on at the ordinary temperature very much more slowly than camphor, and the products of the action are different, for on pouring into water to get rid of the excess of phosphorus pentachloride and oxychloride, the authors obtained a crystalline compound of the formula $\text{C}_{10}\text{H}_{14}\text{ClPO}(\text{OH})_2$, which they name chlorofenchone-phosphoric acid, and an oil consisting of unchanged fenchone and a substance containing chlorine, probably chlorofenchone.

Chlorofenchone phosphonic acid is a white crystalline solid, melting at 196°. It is very soluble in ether, alcohol, chloroform, and benzene, but more sparingly soluble in water. It is a dibasic acid, and the sodium salt crystallises in white needle-shaped crystals. The lead, barium,

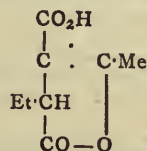
and copper salts are insoluble in water. The oil containing chlorine is at present under investigation.

110. "*Ketolactonic Acid and its Homologues*." By C. H. G. SPRANKLING, B.Sc.

In 1882, Young (*Trans.*, 1883, xliii., 172) observed that when β -ethylacetosuccinic ether is slowly distilled, a little alcohol is liberated, and on hydrolysis of the distillate with hydrochloric acid a crystalline acid, $\text{C}_8\text{H}_{10}\text{O}_4$, is formed in addition to α -ethyl- β -acetopropionic acid and a small quantity of ethylsuccinic acid.

The barium salt, $\text{Ba}(\text{C}_8\text{H}_9\text{O}_4)_2$, is obtained by the action of barium carbonate; a cold solution of barium hydrate gives the salt of β -ethylacetosuccinic acid, whilst at 100° barium carbonate is precipitated and the salt of α -ethyl- β -acetopropionic acid is formed.

From its composition, method of formation, and behaviour it was concluded that the crystalline acid, to which the name ketolactonic acid was given, has the constitution—



At Prof. Young's suggestion, these experiments have been repeated, and a much larger yield of the crystalline acid has been obtained by prolonged heating of the β -ethylacetosuccinic ether before hydrolysis.

The lower homologues of the acid have also been prepared in a pure state from acetosuccinic ether and β -methylacetosuccinic ether respectively, and it has been found that by prolonged heating of β -isopropylacetosuccinic ether and subsequent hydrolysis with hydrochloric acid a very small quantity of the higher homologue is formed.

It is thus shown that the crystalline acid obtained by Young is the third member of a series to which the general name ketolactonic acid may conveniently be given. It will be necessary, however, to call the lowest member of the series ketolactonic acid, the others being named methyl, ethyl, isopropylketolactonic acid.

Ketolactonic acid, $\text{C}_6\text{H}_6\text{O}_4$, does not crystallise; methylketolactonic acid, like the ethyl-compound, forms colourless crystals, m. p. 176°.

The barium salts corresponding to those derived from the ethyl-compound, were prepared from ketolactonic acid and methylketolactonic acid.

The rate of action of sodacetosuccinic ether on the brominated fatty ethereal salts, the rate of elimination of ethyl alcohol from the β -alkylacetosuccinic ethers, and the rate of hydrolysis of the ethers differ greatly in the four cases examined, the rate in general diminishing rapidly with rise of molecular weight.

The hydrolysis of the β -alkylacetosuccinic ethers may take place in two ways—(a) The acetyl-group is replaced by hydrogen and an alkylsuccinic acid is formed; (b) carbon dioxide is evolved and an α -alkyl- β -acetopropionic acid formed. With the ethers investigated, the higher the molecular weight of the alkyl-group the larger is the relative yield of the alkyl succinic acid.

PHYSICAL SOCIETY.

Ordinary Meeting, November 12th, 1897.

Mr. G. JOHNSTONE STONEY, Vice-President, in the Chair.

Mr. J. ROSE-INNES read a paper on "*The Isothermals of Ether*."

The well-known generalisations of Boyle and Gay-Lussac with regard to the pressure, volume, and temperature relations of gases, were examined by Ramsay and

Young, who deduced the law $p = bt - a$, *i. e.*, that pressure is a linear function of temperature, at constant volume, where b and a are functions of volume only. It yet remains to discover the form of these two functions, b and a . The author finds b and a for a large number of volumes, and from them devises an empirical formula. As a preliminary step he examines whether any single algebraical expression can represent the case, so as to determine the probability of discontinuity. For this purpose a graphic method is applied. By plotting $(aV^2)^{-1}$, against $V - \frac{1}{2}$, a curve is obtained of "cusp"-shape. The point of the cusp occurs very near critical volume; it suggests discontinuity in the slope of $(aV^2)^{-1}$. The author concludes that there is extremely rapid change of behaviour of the gas at this point. Again, it is known that the temperature at which pressure is accurately given by the laws of a perfect gas at a particular volume is constant for large volumes until critical volume is approached. The author observes that at the critical volume this temperature diminishes somewhat from its value for large volumes. These conclusions were embodied in a previous paper, and an algebraical expression for pressure in terms of temperature and volume were then given for isopentane. In the present paper the author investigates a similar formula for ether.

Prof. RAMSAY said that experimental errors might account for some of the lack of agreement between proposed formulæ and direct observation of the behaviour of gases. Isopentane was probably a better investigated body than ether, for it was simpler. Ether tended to form complex molecular groupings, but isopentane was probably a mono-molecular liquid.

Prof. PERRY did not quite agree with the author's conclusions. It was necessary to distinguish between a formula founded on a physical hypothesis and a mere empirical formula. The author had assumed that the Ramsay and Young formula was very exact; its originators did not put it forward as being infinitely exact. Probably the best test for such a formula as that under discussion would be derived from some thermo-dynamical conclusion deduced from it. The Rose-Innes formula with five constants and implying discontinuity, was to be distrusted, for there was no such thing as discontinuity in the problem. In any case, an empirical formula should have a very simple form.

Mr. ROSE-INNES admitted that a formula founded on sound hypothesis was to be preferred to empirical expressions. But mathematicians had not yet provided a hypothesis applicable to a substance whose molecular arrangement was so complicated as that of ether. Mathematicians must therefore improve their methods before working formulæ could be deduced from their hypotheses. The use of an empirical formula with five constants was justified by Kepler for the planetary orbits. Kepler used that formula with no other justification than his experience that an ellipse fitted his observations better than a circle. Similar instances might be cited from recent work on the theory of solution, and osmotics.

Mr. JOHNSTONE STONEY was disposed to look for a mathematical cause for the cusp; it was improbable that the physical change was so abrupt as that represented graphically by the author. The question might be tested by plotting the two curves $y = V - \frac{1}{2}$ and $y = aV^2$, and by observing whether these also suggested discontinuity.

Mr. W. L. WATERS then read a paper on the "*Variations in the E.M.F. of the H-form of Clark Cells with Temperature.*"

The authors, Messrs. F. S. Spiers, F. Twyman, and W. L. Waters, have investigated how nearly the true E.M.F. of Clark cells can be computed at different temperatures by applying the ordinary temperature correction. As a standard, two cells of the Muirhead type are employed. The four cells under test could be put through cycles of temperature in a special heating-bath, containing oil circulated by a centrifugal pumping-

vane. E.M.F.'s were determined by a potentiometer method, and a careful study was made of the "lag" of E.M.F. behind temperature. The results are given in the form of curves. It is shown that "lag," in the H-form of cell, is less than in the "Board of Trade" form. Under ordinary conditions, when the rate of variation of temperature is less than 2° C. per hour, by applying temperature-corrections the true E.M.F. of the H-form can be found to within a ten-thousandth of a volt. In this respect there is little to choose between the H-form and the "Muirhead" cell.

Mr. W. R. COOPER thought the authors did not express the case clearly. The E.M.F. of the "Board of Trade" cell could not, with reason, be itself stated within 1 per cent. But in some cases, when for instance cells were used differentially, greater accuracy might be required, as for example when a constant source of E.M.F. was being compared with the variations of another source; here it might be necessary to know the "lag." He would like to know with what degree of accuracy the E.M.F. of the standard cell was determined by the authors. The lag that occurred in the "Board of Trade" cell was probably due to diffusion, crystallisation, and solution.

Mr. WATERS said the E.M.F. of the standard was measured by a Kelvin balance, to one in ten-thousand.

The VICE-PRESIDENT proposed a vote of thanks to the authors, and the meeting adjourned until Nov. 26th.

THE CHEMICAL AND METALLURGICAL SOCIETY OF SOUTH AFRICA.

Meeting held August 21, 1897, at Johannesburg.

Mr. CHAS. BUTTERS, President, in the Chair.

AFTER the election of several new members, the meeting proceeded to discuss a number of amendments to the by-laws of the Society, principally with regard to the reduction of the subscription and widening the qualifications for membership. After the general business had been disposed of, a special meeting was held at which the amendments were all carried unanimously.

The meeting proceeded to discuss the President's Address.

Mr. J. R. WILLIAMS paid a well-deserved compliment to the ability of the President and the excellence of his address, but asked for an explanation of one or two points concerning the roasting and cyaniding of the ore.

Mr. CROSSE confirmed Mr. Williams's experience of having obtained poor results from extracting with cyanide, even after partial roasting; Barberton ore contained 30 to 50 per cent of pyrites, and the results from cyaniding were very poor.

After a few remarks from other gentlemen with regard to the effect of arsenic, the debate on Mr. Williams's paper on the "*Treatment of Battery Slimes*" was opened.

Mr. FRANKLIN WHITE, who spoke at some length, criticised the figures given as to the percentage efficiency of treating the slimes, and pointed out that Mr. Williams distinctly hinted that the number of tons treated was less than 6643, though that figure was used in calculating the cost out to the hundredth of a penny, and further, that by an error on the part of the accountant, the cost of working is arrived at by using the total number of tons crushed as a divisor, viz., 54,531, instead of 21,539, the actual weight of slimes; this of course, he contended, introduces a serious error.

In reply, however, Mr. WILLIAMS ridiculed Mr. White's contention, and claimed that his method of calculation was correct.

Mr. CALDECOTT pointed out that dry crushing and direct cyanide treatment is largely practised in New Zealand and America, and that there was in the Transvaal, though not on the Randt, a Company using this method at a total cost of only ten shillings per ton.

A few remarks were then made by Mr. Cross on Mr. Caldecott's paper on "The Solution of Gold in Accumulated and Other Slimes," the further discussion of which was postponed to the next meeting.

Meeting held September 18, 1897.

Mr. CHAS. BUTTERS, President, in the chair.

In the further discussion of Mr. Williams's paper on the "Treatment of Battery Slimes," the point was raised as to the most efficient method adopted for stirring the slimes, either by mechanical stirrers or by the introduction of air.

The PRESIDENT was of the opinion that a larger volume could be agitated at a less cost by using paddles than by air; but he thinks there is possibly a field for the younger men who adopt agitation solely by air. The time necessary for agitation depends entirely on whether the material is fresh or old, and whether it contains much reducing matter; at the City and Suburban it is as high as 18 hours, but they are not even then quite satisfied with their gold solution.

Mr. MACINTYRE, who has been treating slimes at Spitzkop for about six months, found that by increasing the number of blades in the mechanical stirrers the cone towards the centre was entirely got rid of; the time for treatment was about two days, and in a small plant about 15 tons a day were treated.

After a few remarks from one or two other gentlemen the further discussion on this paper was adjourned, and the discussion on Mr. Caldecott's paper on "The Solution of Gold in Accumulated and Other Slimes" was resumed.

Mr. CROSSE referred to the variation in the specific gravity of different slimes, five samples of which were as follows:—Robinson, 2.38; Meyer and Charlton, 2.59; Goldenhuis Estate, 2.31; Lancaster, 2.50; and Bonanza, 2.62. This is an important subject, as, for the purpose of calculating the amount of slimes in the liquid, the specific gravity will have to be determined before the necessary factor can be applied.

The PRESIDENT, in referring to the use of air, said he considered it as one of the most important discoveries we have had in connection with the cyanide process.

Dr. LOEY considered that the question of the succession in which oxidation takes place in pyritic ores was an open one, and will require more discussion and experimenting on before it can be decided.

This concluded the discussion, and Mr. Caldecott will reply at the next meeting.

Mr. CROSSE then read a paper entitled "Some Notes on Assaying Ground Graphite Crucibles." What is needed for this assay is a reagent that will give enough oxygen to burn away all the graphite, and then lose its excess of oxygen, so that a nearly neutral body is left. Mr. Crosse finds that powdered dioxide of manganese satisfies the required conditions. He mixes 10 grms. of the sample with 35 grms. of the dioxide in a H crucible and raises the temperature to a bright red heat; he then reduces the temperature and adds a mixture of—

- 100 grms. flux (carb. potash 2 parts, borax 1, salt 1).
- 50 " litharge.
- 2 " flour.
- 20 " silica.

The whole fuses together perfectly: when finished the crucible is cooled and broken open, and a soft lead button is obtained containing all the gold.

During the discussion which followed this paper the PRESIDENT introduced some notes on the by-products from the gold industry, viz., from the mills, the chlorination works, the cyanide works, and the melting room, and after thoroughly treating the subject he recommended that the attention of managers and directors should be given to these sources of income, remarking that frequently the battery is utilised as the great sewer through which these valuable products disappear.

CORRESPONDENCE.

THORIUM ACETYL-ACETONATE.

To the Editor of the Chemical News.

SIR,—In reply to "Allanite," no doubt his question will be answered by M. G. Urbain, the author of the article on "Thorium," but, if not, it will please "Allanite" to learn that acetylacetone is not ordinary acetone. Acetylacetone, $\text{CH}_3\text{—CO—CH}_2\text{—CO—CH}_3$, is prepared by either of two methods, viz.,—(1) by the action of Al_2Cl_6 upon acetyl chloride; (2) by the action of Na upon a mixture of aceto-acetic ether and "ordinary" acetone. It is a liquid with a b.p. 137° .

The works he refers to in his letter are too old, but it is described in Bernthsen, p. 238.

Hoping now he will get over his difficulty—I am, &c.,

J. A. FOSTER, A.I.C.
Assist. Admiralty Chemist.

H.M. Dockyard, Portsmouth,
November 13, 1897.

WIRE GAUZE.

To the Editor of the Chemical News.

SIR,—No doubt many of your readers, like myself, have had great trouble with their wire gauze for the tops of burners and stands. I have tried many different kinds, and find that ordinary nickel steel gauze, costing about threepence a square foot, is very satisfactory. It does not rust or perish to anything like the extent that other gauzes do.—I am, &c.,

H. L. ROBINSON.

Chemical Laboratory, Vickers, Sons, & Maxim,
Erith, Kent, Nov. 14, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 18, November 2, 1897.

Preparation and Properties of Calcium, Strontium, and Barium Borides.—H. Moissan and P. Williams.—The authors arrive at the following conclusions:—The three alkaline earthy metals, calcium, barium, and strontium, yield with boron compounds of the formula B_6R , a formula identical with that of the nitrides of Curtius. These compounds are perfectly crystalline; they scratch ruby, possess a great stability, do not decompose cold water as do the carbides, and are especially destroyed by oxidising agents; they are not comparable to the alkaline-earthly carbides and silicides in composition and in properties.

On the Atomic Weights of Argon and Helium.—H. Wilde.—This paper will be inserted at some extent.

On Stannic Acids.—R. Engel.—The results of the author's researches may be summarised as follows:—Pure metastannic acid, isolated from a metastannate or from metastannyl chloride, and dried in a dry vacuum, has the composition $(\text{SnO}_2)_{5/5}\text{H}_2\text{O}$ assigned to it by Fremy in his second study. It contains about 11 per cent of water (or theoretically 10.7).

The Use of Fluoresceine for the Detection of Traces of Bromine in a Saline Mixture.—H. Baubigny.

The Crystallographic Identity of Dextro-rotatory and of Levo-rotatory Asparagine.—P. Freundler.

A Study of the Transformation of Saccharine Matter into Oil in Olives.—C. Gerber.—Olives present a quotient superior to unity when the proportion of mannite decreases and that of oil increases. This quotient is due to the formation in the oil itself of more olive oil at the expense of the mannite.

MISCELLANEOUS.

Royal Institution.—Professor Oliver Lodge, F.R.S., will deliver the first of a Course of Six Christmas Lectures (specially adapted to young people) on "The Principles of the Electric Telegraph," at the Royal Institution, on December 28th. The remaining Lectures will be given on Dec. 30, 1897, and January 1, 4, 6, 8, 1898.

Imperial Institute.—The Winter Course of Lectures at the Imperial Institute will be opened on Friday, November 19th, at 9 p.m., when His Royal Highness the Prince of Wales, President of the Institute, will occupy the chair at an illustrated Lecture by Mr. F. G. Jackson, F.R.G.S., leader of the Jackson-Harmsworth Expedition, entitled "Three Years in the Arctic."

On Monday, the 22nd of November, at 8.30 p.m., Mr. E. S. Bruce, M.A., will read a paper on "Electric Balloon Signalling applied to Scientific Exploration in Arctic and Antarctic Expeditions," fully illustrated by working models and experiments, at which Major P. A. MacMahon, R.A., F.R.S., will preside.

On Monday, November 29th, at 8.30, Sir George Scott Robertson, D.C.L., will deliver an illustrated Lecture on "The Wild Kafirs of the Hindu Kush."

On Monday, December 6th, an illustrated Lecture on "The Mineral Resources of British Columbia and the Yukon" will be given by Mr. A. J. MacMillan, of Rossland, B.C., formerly British Agent for the Government of Manitoba, who has been specially supplied with specimens, &c., to illustrate this Lecture, by the Government of British Columbia. The Hon. Forbes G. Vernon, Agent-General for British Columbia, will take the chair.

On Monday, December 13th, Professor W. C. Roberts-Austen, C.B., F.R.S., will Lecture on "Canada's Medals"; the Right Hon. Lord Strathcona and Mount Royal, G.C.M.G., High Commissioner for Canada, will preside.

On Monday, December 20th, Mr. Boverton Redwood, F.R.S.E., will Lecture on "The Petroleum Sources of the British Empire."

Of the above Lectures, those on the 6th and 13th of December, by Mr. A. J. MacMillan and Professor Roberts-Austen respectively, will be open free to the public (seats being reserved for Fellows of the Institute); the others are open only to Fellows of the Imperial Institute and persons introduced by them.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Calcium Sulphate.—Will any reader kindly inform me whether there is any considerable demand for crude calcium sulphate and the approximate price per ton.—B.

Finishing Woollen Goods.—I am anxious to ascertain a simple and inexpensive mode of washing and finishing large quantities of woollen goods without them shrinking. I should therefore be much obliged if you could put me in communication with any person having this special knowledge.—G. W. WALKER, Springfield Bleach Works, Bulwell, Nottingham.

MEETINGS FOR THE WEEK.

WEDNESDAY, 24th.—Society of Arts, 8. "Progress of Metallurgy and Metal Mining in America during the Last Half Century," by Prof. James Douglas.

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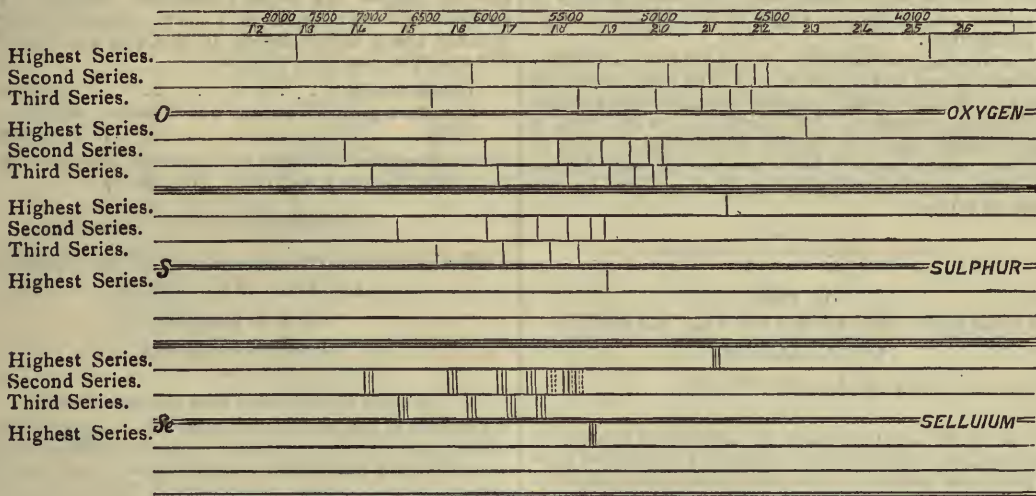
VOL. LXXVI., No. 1983.

ON THE SPECTRA OF OXYGEN, SULPHUR, AND SELENIUM.*

By C. RUNGE and F. PASCHEN.

Two years ago Prof. Pasteur and I showed that the spectrum of helium consisted of six so-called series, which may be arranged in two sets of three, each set resembling very closely the spectrum of one of the alkali metals. From this fact we drew the conclusion that helium probably consisted of two elements, one element corresponding to each of the two sets. Since then we have made an investigation of the spectrum of oxygen as it is exhibited by the electric current passing through a vacuum tube containing oxygen, when no spark gap or Leyden jar is interposed in the circuit; and we have found that this spectrum

row contract, the second one on the right being much narrower than the one on the left. Secondly, whereas in the triplets of the second and third row the strongest line has the lowest wave-number, and the weakest line the highest, this order is reversed in the triplets of the first row. This is in perfect accordance with the spectra of the alkalis. There we have doublets instead of triplets; but we find the same two circumstances. Whereas in the so-called secondary series, corresponding here to the second and third row, the difference of wave-numbers remains the same, it grows smaller with increasing wave-number in the so-called principal series corresponding to the first row. Secondly, whereas in the secondary series the stronger line has the lower wave-number, this order is reversed in the principal series. This is connected with a law discovered by Rydberg. Rydberg showed that in the spectra of the alkalis the difference between the wave-numbers of the common limit of the two secondary series and the wave-number of the limit of the principal series is equal to the wave-number of the first member of the principal series. Now there are two limits of the secondary series, one for the stronger lines and one for the weaker lines of the doublets; but there is only one limit of the principal series. Therefore there are two differences, a, b , between the limits of the secondary series and the limit of the principal series, a corresponding to the limit of the



of oxygen closely resembles that of helium. You see a drawing of it here, together with the spectra of sulphur and selenium plotted to the scale of wave-numbers, 20, for instance, corresponding to the wave-length $1/20,000$ c.m., equal to 5000 Angström's units.

But this is only a very crude representation of what is actually seen. The drawing only gives the lines, not their intensity, which decreases with the increasing wave-number. Neither does it render the beautiful details of the lines giving an untiring sense of pleasure to the eye. The lines of the second and third row, for instance, are triplets of very characteristic appearance,—so characteristic that Piazzesi Smyth, who was the first to observe some of them, very justly remarks that it is as easy to recognise one of them in the spectrum of a vacuum tube swarming with impurities as it is to pick out from among a crowd of civilians a soldier with scarlet coat and cross-belts. The first row also consists of triplets; but they differ in two very remarkable ways from the triplets of the second and third row. Whereas here the differences of wave-numbers are the same for all the triplets, the triplets of the first

stronger lines, b to that of the weaker lines. Now as the principal series overlaps the secondary series, a is larger than b , and therefore the first member of the principal series consists of a doublet with the wave-numbers a and b , the larger number a corresponding to the stronger line. Rydberg had the boldness to predict that his law would hold good for triplets, although principal series of triplets had not yet been observed; and he was right. We see his law obeyed in the spectrum of oxygen, and similarly in that of sulphur and selenium.

I cannot enter into the details of the mathematical formulæ representing the wave-numbers of each series, and proving the analogy of the three series of each set with the three series of each of the alkali metals. But what Professor Paschen and I wish to convey is this:—*There is about as much spectroscopic evidence for the duplicity of oxygen that there is for the duplicity of helium. Therefore, if on chemical reasons we are not allowed to consider oxygen as consisting of two elements, then spectroscopy does not give us any reason to believe in the duplicity of helium.*

At the same time we have investigated the spectrum of sulphur and of selenium, two bodies chemically related to oxygen, and we have found that under analogous conditions

* Read before the British Association (Section A), Toronto Meeting, 1897. See also *Wied. Ann.*, Bd. 61, 1897.

both emit a spectrum very similar to the set of the first three series. Whether the three other series also find their analogy we do not venture to contend. There is, however, a strong line in the spectrum of sulphur, as well as in that of selenium, that seems to correspond to the strong violet oxygen line.

The three series of the spectrum of sulphur and of selenium are triplets of the same build as the oxygen triplets; only they are wider, the difference of wave-numbers being, roughly speaking, proportional to the square of the atomic weights, a law that also holds good for several other groups of chemically related elements.

The spectrum of oxygen has been partly observed by other spectroscopists. Piazzzi Smyth and A. Schuster should be named in particular; but these spectra of sulphur and selenium have now been observed for the first time. It is strange that it should be possible to find new spectra of bodies so well known as sulphur and selenium; and we can perhaps infer from this fact that many of the lines which astronomers observe in the spectra of the sun and of the stars, the origin of which is not known, may after all belong to well-known elements.

The spectrum of sulphur as a whole consists of lines of smaller wave-numbers, that is to say of slower oscillations, than that of oxygen; and the spectrum of selenium consists, again, of slower oscillations than that of sulphur. This is what we should expect from the atomic weights of the substances, the heavier atoms oscillating slower than the lighter ones.

In analysing the radiations emitted by the elements, spectroscopy can as yet give us very little insight into the nature of these complex, variable, and mysterious things that science pleases to call elements; but we think a time will come when the regular distribution of lines in what we call series will find a mechanical explanation. And that, we think, will shed a flood of light on the nature of things.

ON THE IMPURITIES OF COMMERCIAL CARBIDES OF CALCIUM.

By H. LE CHATELIER.

THE study of the impurities of carbide of calcium is interesting on account of the indications it can give as to the reciprocal affinities of certain bodies when heated to a temperature of about 2000°.

After calcium and carbon, the two most abundant elements present are silicon and iron. It is possible that definite combinations of either of these bodies may exist with each of the other three; which are those, we may ask, which are formed by preference at the temperature of solidification of calcium carbide?

The iron, which is the least abundant of the four elements under consideration, might be entirely saturated by one or the other of the remaining three. As a matter of fact it is combined exclusively with the silicon. It is detected by treating the hydrated carbide with an acid, and suspending the insoluble residue in iodide of methyl. Small crystals of silicide of iron are precipitated; these have been studied by M. Moissan; by analysis they have been proved to be SiFe_2 .

The excess of silicon combines either with the carbon or with the calcium, according to the relative proportions of these two bodies; if the quantity of carbon present is in excess of the calcium, a silicide of carbon is formed, crystallising in apparently hexagonal plates, and generally of a blue colour. It is found, with the excess of graphite, floating on the surface of the iodide of methyl. If, on the contrary, the quantity of calcium is in excess of the quantity of carbon present, a silicide of calcium is formed, which is disseminated throughout the mass of

the carbide in the form of metallic grains, having the colour and lustre of zinc. These can be isolated by rapidly quenching the carbide in a large excess of cold water, separating by levigation the coarser residues, and washing them for a few moments with a solution of acetic acid. The final residue is composed of silicide of iron, and of the coarsest grains of silicide of calcium, which have withstood the rapid washings intended to isolate them. In such a case there would be neither graphite nor silicide of carbon present, for this reason, that the calcium must be in excess to allow of the formation of silicide of calcium. In fact, the determining affinities which govern the combination of these elements are those of iron for silicon, and of calcium for carbon. These are first satisfied, and the remaining bodies combine amongst themselves in a manner varying according as one or the other is in preponderating proportion.

There seem to exist two distinct silicides of calcium; one of them is hardly attacked by nitric acid, but is, on the contrary, easily attacked by hydrochloric acid, with the formation of an insoluble yellow matter, called *silicone* by Wöhler. The other, easily attacked by nitric and acetic acids, gives, with hydrochloric acid, a deposit not yellow but white, which, like *silicone*, is soluble in potash, with an abundant disengagement of hydrogen.

In the attack of silicide of calcium we more frequently get the yellow and white compounds together. The analyses of these mixtures lead one to suspect compounds between those corresponding to $\text{Si}_2\text{O}_4\text{H}_4$ and $\text{Si}_2\text{O}_3\text{H}_4$. For example, one analysis gave—

Yellow matter	0.52 grm.
Hydrogen given off (0° and 760 m.m.) ..	630 c.c.
Silica	0.57 grm.

which corresponds exactly to the second formula.—*Bull. Soc. Chim.*, Series 3, xvii.-xviii., Nos. 16, 17.

THE COMBUSTION OF ORGANIC SUBSTANCES IN THE WET WAY.*

By I. K. PHELPS.

(Concluded from p. 247).

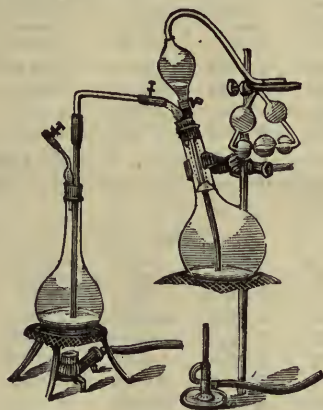
Method of Oxidation with Chromic Acid.

A CONCENTRATED mixture of chromic and sulphuric acids, although a much more powerful oxidiser than potassium permanganate in aqueous solutions, fails to oxidise completely many organic compounds. Thus, Cross and Higgin (*Journ. Chem. Soc.*, 1882, 113) have shown that carbohydrates are among the number of organic substances; later, Cross and Bevan find that carbohydrates and many other substances are oxidised completely to a mixture of carbon dioxide and monoxide. Messenger (*Ber.*, xxiii., 2756) has proven that carbon may be determined in organic compounds by passing the mixed products, resulting from the oxidation with chromic and sulphuric acids, through a short combustion-tube filled with granular copper oxide and heated in a furnace—all of which facts have been confirmed in my own experience.

Ludwig (*Am. Chem. Pharm.*, clxii., 47) has observed that the contact of carbon monoxide with a mixture of chromic and sulphuric acids, especially when hot, results in the oxidation of that gas to carbon dioxide. This fact suggested the idea of substituting for the apparatus described above a new form, adapted to retain the first products of oxidation in prolonged contact with the oxidising mixture. This apparatus, shown in the accompanying figure, by means of which the sequel shows, it has been found possible to extend the availability of the oxidising mixture, is put together as follows:—A

* Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, Series 4, Vol. iv., No. 23, November, 1897.

thick-walled round bottomed flask of a litre's capacity, serving as an oxidising chamber, was closed by a rubber stopper with two perforations, through one of which passes the tube of a separating funnel of about 100 c.m.³ capacity. The tube of this funnel reached nearly to the bottom of the flask and is drawn out at the lower end. A disc of platinum foil is hung in the neck of the flask, nearly closing it, and held in place by a platinum wire passing through the foil and tucked under the rubber stopper where the funnel tube enters. The second hole of the stopper is filled by the exit tube, a glass tube of 0.7 c.m. internal diameter. This tube is expanded just above the stopper to a small bulb which serves to prevent mechanical loss of the solid contents of the flask during the boiling. This tube is joined by means of a rubber connector (provided with a screw pinchcock) to the inlet-tube of the absorption flask, which is an ordinary 500 c.m.³ round-bottom flask. This flask is also closed by a rubber stopper with two perforations, through one of which passes the inlet-tube described above and through the other the exit tube, which is also enlarged to a small bulb just above the stopper and is closed by a rubber connector and screw pinchcock. The glass ground stopper of the funnel tube is carefully cleaned and lubricated with a thick solution of metaphosphoric acid,



Instead of getting the vacuum by the water-pump, it may be gotten almost as quickly and certainly more simply by boiling water in the evolution flask and the barium hydroxide solution in the absorption flask at the same time—both flasks being connected ready for making a determination. When steam issues in good quantity from the exit tube of the absorption flask, the burner is removed from under the evolution flask and its screw pinchcock closed, and then the burner under the absorption flask and its screw pinchcock also quickly closed. The flasks are then allowed to cool.

In making a determination, the organic substance is weighed out in a counterbalanced bulb, so thin that it may be easily broken later, and made with a wide mouth for convenience in introducing the solid substance. After the substance is weighed, the mouth of the bulb is sealed by heating in a small blowpipe flame, and the tube introduced into the evolution flask, together with an amount of pure potassium dichromate, which is known to be in excess of that required to oxidise the organic substance. The flasks are connected, as already described, with an appropriate amount of barium hydroxide solution in the absorption flask and 10 c.m.³ of pure water in the evolution flask, and the vacuum obtained (as described above) by boiling both flasks, the boiling being stopped when the water in the evolution flask has decreased to 2 or 3 c.m.³. Naturally, this boiling must be so regulated as not to allow loss of the solid material in either flask. The vacuum obtained, the tube containing the organic substance is broken by shaking the flask, and 20 c.m.³ of con-

centrated sulphuric acid, previously purified from organic material by heating to the fuming point with a few crystals of potassium dichromate, are run in through the funnel tube, when reduction of the chromic acid soon becomes evident. While still hot, the acid is shaken in the flask violently, the platinum foil hung in the neck serving to protect the rubber stopper. The flask is warmed to approximately 105° C., the highest temperature to which, as shown by Cross and Bevan (*Journ. Chem. Soc.*, liii., 889), a mixture of chromic and sulphuric acid may be safely heated without the disengagement of oxygen gas. Water is then run in until the crystals of chromic anhydride have disappeared and the danger of the evolution of oxygen is passed. The solution is heated to its boiling-point, care being taken that it shall not get under pressure, which can easily be observed by opening momentarily the stopcock of the funnel tube and noting the direction of the flow of water contained in the funnel. The flask is shaken and heated alternately for five minutes—a period of time which appears to be sufficient to bring about the oxidation of the small amount of carbon monoxide originally produced. Then more water (60 to 70 c.m.³) is introduced through the funnel and the stopcock between the boiling and absorption flasks opened, when the carbon dioxide enters the absorption flask, which is kept cool and shaken as before. The contents of the evolution flask are then heated to boiling and a slow current of air, freed from carbon dioxide by passage through potash bulbs, allowed to enter through the funnel tube to keep the liquid from undue bumping. The boiling is continued for fifteen minutes, after which the excess of barium hydroxide is determined iodometrically, and thus the carbon dioxide present estimated as before. Table IV. shows results obtained by the treatment of crystallised ammonium oxalate and cane-sugar, re-crystallised from dilute alcoholic solution, in this manner.

TABLE IV.					
Substance taken. Grm.	BaO ₂ H ₂ taken. Grm.	BaO ₂ H ₂ found. Grm.	CO ₂ found. Grm.	CO ₂ calculated. Grm.	Error on CO ₂ . Grm.
<i>Analysis of Ammonium Oxalate.</i>					
0.5009	1.3534	0.1469	0.3097	0.3101	0.0004-
0.5006	1.3400	0.1308	0.3103	0.3099	0.0004+
0.5005	1.3400	0.1343	0.3094	0.3098	0.0004-
1.0002	2.5460	0.1347	0.6188	0.6192	0.0004-
1.0010	2.5192	0.1094	0.6185	0.6197	0.0012-

<i>Analysis of Cane-sugar.</i>						
1.	0.2001	1.3926	0.1905	0.3085	0.3088	0.0003—
2.	0.2000	1.3926	0.1936	0.3077	0.3086	0.0009—
3.	0.2001	1.3926	0.1857	0.3097	0.3088	0.0009+
4.	0.2014	1.3400	0.1279	0.3111	0.3108	0.0003+

The results are evidently very satisfactory.

The Determination of the Oxygen required to Oxidise an Organic Substance.

Several different methods have been proposed for estimating the oxygen present in organic substances, depending in general upon the determination of the oxygen which must be supplied to burn the substance to a known amount of carbon dioxide and water—thus discovering by difference the oxygen originally contained in the substance. Lavoisier is said to have measured directly the oxygen used in burning organic substances; Gay-Lussac and Thénard determined the oxygen used by measuring the amount of potassium chlorate reduced by burning the organic compound; Baumhauer (*Ann. Chem. Pharm.*, xc., 228) determined the oxygen used by measuring the volume of oxygen entering the combustion furnace and subtracting the measure of the gas coming from the combustion tube, which was set up according to the well-known method for determining carbon and hydrogen; Stromeyer (*Ann. Chem. Pharm.*, cxvii., 247) determined the amount of copper reduced by the ignition

of the substance in copper oxide; Ladenburg (*Ann. Chem. Pharm.*, cxxxv., 1) oxidised the substance by heating in a sealed tube with a known amount of iodic acid, determining at the end of the operation the amount of iodic acid left; Mitscherlich (*Pogg. Ann.*, cxxx., 536) has estimated the oxygen in organic substances directly by decomposing the substance by ignition in a stream of chlorine gas, estimating the oxygen content by determining the resulting carbon dioxide and monoxide.

As it has been shown in the work described that carbon may be determined in organic substances by oxidation with chromic and sulphuric acids without the evolution of oxygen gas, it would seem that the determination of the oxygen in the substance might be effected by determining the amount of chromic acid used in the operation, taking into consideration the products of combustion. This can be readily accomplished by taking a weighed amount of pure potassium dichromate as the oxidising agent and determining, at the end of the operation, the amount of chromic acid left by treatment of the residue with hydrochloric acid, absorption of the chlorine evolved in an alkaline arsenite of known strength, and titration of the excess of that substance with decinormal iodine solution.

To test the accuracy of the determination of chromic acid under these conditions of analysis, weighed portions of pure fused potassium dichromate were introduced into a Voit flask, whose outlet tube was sealed to the inlet tube of a Drexel wash bottle, the outlet of which, in turn, was sealed to a Will and Varrentrapp absorption apparatus. An amount of hydrochloric acid, more than enough to completely reduce the chromate (15 to 40 c.m.³ of the strongest acid), was added with 20 c.m.³ of strong sulphuric acid, and the total volume made up to 120—140 c.m.³ of liquid. The sulphuric acid used here was purified from carbonaceous matter (as in the carbon determination above) by heating with a few crystals of potassium dichromate, the excess of which was reduced by holding the acid at a fuming point for about two hours, when a portion diluted with water gave no colour with potassium iodide and starch paste. Pure arsenious oxide, in amount slightly in excess of that required to take up the oxygen to be given up by the chromate, was dissolved by the aid of heat in a solution of pure sodium hydroxide, taken in such quantity as to more than neutralise the arsenious acid and the hydrochloric acid used to reduce chromate, and this solution was introduced into the

Drexel wash bottle. The flask was then connected with the wash bottle, using a thick solution of metaphosphoric acid to lute the joint between the flask and its stopper. The absorption apparatus was charged with a dilute solution of sodium hydroxide. Carbon dioxide was generated in a Kipp generator by the action of hydrochloric acid on marble and purified from reducing matter by bubbling through a strong solution of iodine in potassium iodide, and finally washed with a solution of potassium iodide alone. A slow stream of this purified carbon dioxide was allowed to enter the inlet tube of the Voit flask, the contents of which were then boiled. When a concentration to a volume of 30 to 40 c.m.³ was reached, the boiling was discontinued, and, after cooling and disconnecting the flask, the contents of the receiver were made acid with sulphuric acid and then alkaline with acid potassium carbonate, when the excess of arsenite was determined by titration with decinormal iodine solution. Sometimes during the reduction of the chromic acid, the red fumes of the chlorochromic anhydride volatilised to the receiver; but since the chromic acid thus produced is reduced later by the arsenite (Browning, *Am. Journ. Sci.*, 1896, vol. i., 35), this transfer is of no account in the working of the process. The following results were thus obtained:—

TABLE V.

	K ₂ Cr ₂ O ₇ taken. Grms.	As ₂ O ₃ taken. Grms.	As ₂ O ₃ found. Grm.	K ₂ Cr ₂ O ₇ found. Grms.	Error on K ₂ Cr ₂ O ₇ . Grm.
1.	5'0002	5'1025	0'1144	4'9447	0'0555—
2.	5'0018	5'0799	0'0526	4'9849	0'0169—
3.	5'0005	5'0801	0'0582	4'9782	0'0223—
4.	5'0013	5'0706	0'0908	4'9365	0'0648—

The cause of the error shown in these experiments was traced finally to too great concentration of the sulphuric

TABLE VI.

	K ₂ Cr ₂ O ₇ taken. Grms.	As ₂ O ₃ taken. Grms.	As ₂ O ₃ found. Grm.	K ₂ Cr ₂ O ₇ found. Grms.	Error on K ₂ Cr ₂ O ₇ . Grm.
1.	1'0004	1'0500	0'0398	1'0014	0'0014+
2.	1'0007	1'0531	0'0437	1'0006	0'0001—
3.	2'0013	2'0501	0'0299	2'0026	0'0013+
4.	2'0037	2'0727	0'0502	2'0049	0'0012+
5.	5'0020	5'1002	0'0495	5'0068	0'0048+
6.	5'0037	5'1018	0'0513	5'0066	0'0029+

TABLE VII.

	Substance taken. Grm.	CO ₂ found. Grm.	Error on CO ₂ . Grm.	K ₂ Cr ₂ O ₇ taken. Grm.	As ₂ O ₃ taken. Grm.	As ₂ O ₃ found. Grm.	Oxygen used. Grm.	Oxygen required by theory. Grm.	Error on oxygen. Grm.
<i>Analysis of Ammonium Oxalate.</i>									
1.	1'0122	0'6265	0'0001—	2'0009	1'3002	0'0000	0'1160	0'1139	0'0021+
2.	1'0019	0'6212	0'0010+	2'0002	1'3517	0'0440	0'1147	0'1128	0'0019+
<i>Analysis of Phthalic Acid.</i>									
1.	0'1002	0'2138	0'0014+	2'0012	1'2004	0'0814	0'1456	0'1448	0'0008+
2.	0'1093	0'2324	0'0007+	2'0000	1'1031	0'0634	0'1582	0'1580	0'0002+
<i>Analysis of Cane-sugar.</i>									
1.	0'2025	0'3117	0'0008—	3'0000	1'7002	0'0796	0'2275	0'2273	0'0002+
2.	0'4012	0'6166	0'0024—	5'0000	2'3022	0'0366	0'4495	0'4502	0'0007—
<i>Analysis of Paper.</i>									
1.	0'3034	0'4932	0'0010—	3'5015	1'4017	0'0879	0'3589	0'3598	0'0005—
2.	0'4523	0'7334	0'0033—	5'0035	1'8000	0'0710	0'5368	0'5358	0'0010+
<i>Analysis of Tartar Emetic.</i>									
1.	0'5057	0'2671	0'0009—	2'5018	1'7000	0'0766	0'1459	0'1462	0'0003—
2.	1'0099	0'5321	0'0030—	3'5003	1'7520	0'0198	0'2911	0'2919	0'0008—
<i>Analysis of Barium Formate.</i>									
1.	1'0079	0'3906	0'0006+	3'0026	2'2002	0'0496	0'1423	0'1422	0'0001+
2.	1'5014	0'5814	0'0005+	3'0010	1'8080	0'0890	0'2118	0'2118	0'0000±

acid in the process. When the boiling begins the chromate is reduced gradually and if the evaporation of the water is pushed too rapidly, the sulphuric acid may reach a strength at which it begins to cause the reduction of the chromic acid with the evolution of oxygen instead of chlorine.

The obvious remedy is to conduct the boiling operation more slowly. It was found that, if from five to six hours time was taken for the proper concentration of the contents of the Voit flask, the presence of the sulphuric acid worked no harm, as will be seen from the following results. Experiments 1 and 5 were made with 5 c.m.³ of sulphuric acid present and the others with 20 c.m.³ as used before. (Table VI.).

In applying this method to the determination of oxygen used in the oxidation of an organic substance, the carbon determination was made as already described, the amount of water used being such as to leave 60 to 80 c.m.³ of liquid in the boiling flask after the carbon dioxide had been driven to the absorption flask by boiling. This liquid was then washed into the Voit flask and the chromic acid remaining determined by a second distillation (this time with hydrochloric acid) in the manner described above. In each of the experiments recorded in Table VII., 20 c.m.³ of purified sulphuric acid were used in the carbon determination, and 35 c.m.³ of hydrochloric acid (sp. gr. 1.2) in the chromic acid determination. The ammonium oxalate used was the pure crystallised salt; the phthalic acid was re-crystallised from its water solution and dried for a short time over sulphuric acid; the cane-sugar was selected crystals of rock candy, re-crystallised from dilute alcoholic solution and dried for a long time over sulphuric acid; the paper was ashless filter-paper, dried to a constant weight over sulphuric acid; the tartar emetic was re-crystallised from water solution and air dried; the barium formate was prepared by treating formic acid with an excess of pure barium carbonate, filtering hot, and allowing the product to crystallise.

From these results, it will be seen that the process works with accuracy upon a great variety of organic substances. It was found impossible, however, to determine the elements in bodies which are at the same time volatile and hard to oxidise; for instance, ether oxidises easily to acetic acid but difficultly beyond that stage; although the liquid acid is oxidised vigorously by chromic and sulphuric acids, the gaseous acid is hardly attacked at the temperature used; naphthalene was also found to be volatilised, and hence not attacked, to such an extent as to render its determination by this process valueless.

In conclusion, the author wishes to express his thanks to Prof. F. A. Gooch for many helpful suggestions.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, November 4th, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

MESSRS. Harold W. Harrie, W. R. Lang, W. H. Barlow, and A. V. C. Fenby were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Ernest George Annis, Health Office, Town Hall, Huddersfield; William Ball, 54, Stretton Road, Leicester; Richard Oxley Burland, J.P., Poolstock House, Wigan; Alexander McLean Cameron, Daylesford, Victoria; Owen Aly Clark, 12, Abbey Gate Street, Bury St. Edmunds; Alexander Clarkson, 2, Waveney Crescent, Ballymena, Ireland; Frank Collingridge, B.Sc., Kenmore, Shepherd's Hill, Highgate, N.; James Murray Crofts, B.A., Richleigh, Gloucester; David Crole, Primrose Studios, Wellington Square, Chelsea, S.W.; John Daniell, Council of Education Laboratory, Johannesburg, S.A.R.; Andrew

James Dixon, Dapto, N.S.W.; Robert Hamilton, 11, Ibrox Place, Glasgow; John Harger, B.Sc., Ph.D., The Nook, St. James's Mount, Liverpool; Charles Kelly, Oakmere, Hawarden, Cheshire; Tom Lemmey, B.A., Wellington College, Berks; James Scott MacLaurin, D.Sc., Mount Eden, Auckland, N.Z.; Allen Macmullen, 82, James Street, Dublin; Edward Masters, The Aloes, Hinckley Road, Leicester; John A. Mathews, 4, First Place, Brooklyn, N.Y.; Philip George Gregory Moon, 129, Rosary Road, Thorpe, Norwich; Joseph John Mooney, 34, Easter Road, Edinburgh; James Charles Philip, B.Sc., Ph.D., 16A, Merton Road, Victoria Road, Kensington, W.; Alexander Ferguson Reid, Stair Bridge, Stair, Ayrshire; Ernest Henry Roberts, Hollydale, All-farthing Lane, Wandsworth, S.W.; Edward Sydney Simpson, 34, Pier Street, Perth, West Australia; Robert Francis Woodsmith, 89, Bartholomew Close, E.C.; Frederick William Steel, Tamunua, Navua River, Fiji; Michael Edmund Stephens, Avenue House, Finchley, N.; George Stubbs, Arnside, Hertford Road, East Finchley, N.; Edward Howard Tripp, Ph.D., Kent House, Blackheath Hill, S.E.; John Scriven Turner, 20, Bury Street, Bloomsbury, W.C.; Framjee Khursjee Viccajee, Hyderabad, Deccan, India; Percy John Vinter, Wesley College, Sheffield; Arthur James White, Whinsfield, Barrow-in-Furness.

Sir WILLIAM CROOKES then took the Chair, and of the following papers those marked * were read:—

*III. "On the Properties of Liquid Fluorine." By Professors MOISSAN and DEWAR.

The nearest approach to the properties of the mythical alkali or universal solvent of the alchemist is to be met with in fluorine. The transparent vessels in which it can be manipulated have to be made of some fluoride like fluor-spar, and such vessels are equally difficult to construct and ill-adapted for chemical manipulation. Modern research has, however, revealed the fact that the most powerful chemical affinities are completely suspended by allowing substances to come into contact at very low temperatures, and it appeared possible that even fluorine, which has the most powerful chemical activity of all the elements, might be manipulated in glass vessels under such conditions.

In a paper communicated to this Society entitled "The Liquefaction of Air and Research at Low Temperatures" (*Proc.*, 1895, xi., 221), speaking of fluorine, the author remarked, "This is the only widely distributed element that has not been liquefied. Some years ago, Wallach and Hensler pointed out that an examination of the boiling-points of substituted halogen organic compounds led to the conclusion that, although the atomic weight of fluorine is nineteen times that of hydrogen, yet it must in the free state approach hydrogen in volatility. This view is confirmed by the specific refractive index which Gladstone showed was rather lower than hydrogen. If the chemical energy of fluorine at low temperatures is abolished like that of other active substances, then some kind of glass, or other transparent material not so brittle as calcium fluoride, could be employed in the form of a tube, and its liquefaction achieved by the use of hydrogen as a cooling agent."

The inference that fluorine approached hydrogen in volatility was deduced by Wallach and Hensler from a consideration of the boiling-points of the fluorine derivatives of the benzene series.

The following table—

			I.			
Benzene.	Boiling point.	Difference.		Toluene.	Boiling point.	Difference.
C ₆ H ₆	80°	5°		C ₆ H ₅ CH ₃	111°	5°
C ₆ H ₅ Fl	85°			p-C ₆ H ₄ Fl·CH ₃	116°	
C ₆ H ₅ Cl	132°		47°	p-C ₆ H ₄ Cl·CH ₃	160°	

Aniline.		Benzene.	
$C_6H_5NH_2$.. 183°	} 4° 43°	C_6H_6 80°	} 8° 84°
$p\text{-}C_6H_5F\cdot NH_2$ 187°		$p\text{-}C_6H_5F$ 88°	
$p\text{-}C_6H_5Cl\cdot NH_2$ 230°		$p\text{-}C_6H_5Cl$ 172°	

shows that the substitution of 1 atom of hydrogen in these compounds by fluorine only causes an increase of the boiling-point of from 4° to 5° , whereas chlorine causes an increase of from 45° to 50° . Such a relatively large ratio as 1 to 10 in the increment of boiling-points suggests a great difference in the volatility of the elements fluorine and chlorine in the free state. A further examination of the properties of fluorine compounds, however, showed that the volatility of fluorine was not likely to approach that of hydrogen. This will be apparent from the following table.—

II.			
Methane. (absolute).	Difference.	Ethane. (absolute).	Difference.
CH_4 .. 110°	} 90°	C_2H_6 .. 184°	} 58°
CH_3F 200°		C_2H_5F .. 242°	
CH_3Cl 250°	} 50°	C_2H_5Cl 286°	} 44°
CH_4 .. 110°		$C\cdot H_4$.. 110°	
CF_4 .. 259°	$149^\circ = 4 \times 37^\circ$	$C\cdot Cl_4$.. 351°	$241^\circ = 4 \times 60^\circ$
Aldehyde. B. p. (C.)		Benzaldehyde. B. p. (C.)	
CH_3COH 21°	} -11°	C_6H_5COH 179°	} -18°
CH_3COF 10°		C_6H_5COF 161°	
CH_3COCl 51°	41°	C_6H_5COCl 199°	38°

where it is seen that the substitution of hydrogen by fluorine in methane and ethane raises the boiling-point by 90° and 60° respectively, and that the ratio of the increments of boiling-point in corresponding fluorine and chlorine compounds is now not greater than 1 : 2. The boiling-point of methyl fluoride was calculated from the critical point and vapour pressure of this substance as recorded by Professor Collie (*Trans.*, 1889, lv., 110). It will be noted as a curious fact that the substitution of fluorine in the aldehyde radicle causes a lowering of the boiling-point and not an increase, and that the difference in boiling-point between the chlorine and fluorine substitution body in either series is always between 40° and 50° . These considerations induced the hope that liquid air might give the command of a sufficiently low temperature for the liquefaction of fluorine, and that glass vessels might be used to collect the liquid. This view was supported by a consideration of the melting-points of the halogens and the corresponding critical points deduced by following the suggestions of Clarke (*Am. Chem. Soc. T.*, 1896, xviii., 618) as to these relations. Thus the absolute melting-points of chlorine, bromine, and iodine are respectively 171° , 267° and 388° , and, assuming the same mean difference in melting-point extended to fluorine, then its melting-point would be 64° absolute. Now the critical points of chlorine and bromine are about $2\frac{1}{2}$ times the absolute melting-points, thus giving 149° absolute, or -125° , as the probable critical point of fluorine. This critical value is only a few degrees lower than oxygen, and from this calculation the authors were entitled to assume that the position of fluorine as regards volatility would be somewhere between that of oxygen and nitrogen.

The following research was conducted in the Chemical Laboratory of the Royal Institution, to which Professor

Moissan brought the apparatus for the production of gaseous fluorine with which his name will always be identified, and the authors had the invaluable assistance of Messrs. Lebeau, Lennox, and Heath in the conduct of the experiments.

Fluorine was prepared by the electrolysis of potassium fluoride in solution in anhydrous hydrofluoric acid. The fluorine gas was freed from vapours of hydrofluoric acid by being passed through a serpentine of platinum cooled by a mixture of solid carbonic acid and alcohol. Two platinum tubes filled with perfectly dry sodium fluoride completed the purification.

The apparatus used for liquefying the gas consisted of a small cylinder of thin glass, to the upper part of which was fused a platinum tube. This latter contained in its axis another smaller tube, likewise of platinum. The gas to be liquefied enters by the annular space, passes through the glass envelope, and escapes through the small inner tube. The glass envelope was fused to the platinum tube by which the fluorine was supplied.

The glass cylinder being cooled down to the temperature of boiling liquid oxygen (-183°), the current of fluorine gas passed through the bulb without becoming liquid. At this low temperature, however, the gas has lost its chemical activity, and no longer attacks the glass.

On lowering the temperature of the liquid oxygen by exhaustion, a yellow liquid is seen collecting in the glass envelope, while gas no longer escapes from the apparatus. At this moment the tube by which the gas had been escaping is stopped, so as to prevent air from entering and liquefying, and the glass bulb soon becomes full of a clear yellow liquid, possessed of great mobility. The colour of this liquid is the same as that of fluorine gas when examined in a stratum 1 metre thick. Fluorine thus becomes liquid, according to this experiment, at about -185° .

When the bulb containing the liquid fluorine is lifted above the surface of the liquid oxygen, the yellow liquid begins to boil with an abundant disengagement of gas, having all the energetic reactions of fluorine.

Silicon, boron, carbon, sulphur, phosphorus, and reduced iron, cooled in liquid oxygen and then placed in an atmosphere of fluorine, did not become incandescent. At this low temperature, fluorine did not displace iodine from iodides. However, its chemical energy is still sufficiently great to decompose benzene or oil of turpentine with incandescence. It would thus seem that the powerful affinity of fluorine for hydrogen is the last to disappear. The authors have noticed on some occasions that a current of fluorine gas passed into liquid oxygen gives a flocculent precipitate of a white colour, which quickly settles to the bottom. If this mixture is shaken and thrown on a filter, the substance can be collected. It possesses the curious property of deflagrating with violence as soon as the temperature rises.

A new apparatus (Fig. 1) was constructed similarly to that already described (that is to say, a glass bulb, *b*, fused to a platinum tube, *A*, which contained another similar smaller tube, *D*), but having each of the platinum tubes, *B* and *C*, fitted with a screw valve, in such a manner that at any moment communication—either with the outer air or with the current of fluorine—could be interrupted. This little apparatus was placed in a cylindrical glass vacuum vessel containing liquid oxygen, connected with a vacuum pump and manometer.

On repeating the former experiment with freshly prepared liquid air, instead of oxygen, fluorine easily becomes liquid at -190° C. With liquid oxygen as refrigerant, the liquefaction of fluorine takes place at a temperature corresponding to the evaporation of the oxygen under a pressure of 437 m.m. of mercury.

From these two experiments it results that the boiling-point of fluorine is very close to -187° . This number is identical with Olszewski's boiling-point of argon, so that this seems to be the first example of two gaseous elements boiling at the same temperature. It is a justifiable infer-

ence from the boiling-point that the critical point must be about -120° , and thus, in all probability, the critical pressure is about 40 atmospheres, or less than half that of the critical pressure of chlorine, which is 84 atmospheres. This would make the critical constant for fluorine 4 as contrasted with chlorine, which has the value 5.

The following table gives the boiling-point of the halogens:—

	Absolute temperature.	Difference.
Fluorine	87°	153°
Chlorine	240°	
Bromine	337°	97°
Iodine	460°	
		123°

When the little glass bulb was three-quarters full of liquid fluorine, both the valves were closed, and then a good air-pump caused the liquid oxygen serving as refrigerant to boil rapidly at a pressure of 2.5 c.m. Under these conditions a temperature of -210° is reached, yet the fluorine did not show any sign of solidification, but retained its characteristic mobility. In future experiments it will be interesting to try the rapid ebullition of the liquid fluorine itself. During the repetition of this experiment a slight accident occurred. The screw of one of the valves becoming worn, allowed air to leak into the exhausted bulb. This air was immediately liquefied, and in a few moments two distinct layers of liquid were seen; the upper colourless layer, consisted of liquid air; the lower one, of a pale yellow colour, being fluorine.

To prevent the possible ingress of any air, the fluorine was introduced in its liquid state into a glass tube, the end of which was then sealed before the blowpipe. The sealed tube, containing the liquid fluorine, was kept for a long time at -210° by the rapid evaporation of a large quantity of liquid air, but it gave no trace of a solid body.

To determine the density of liquid fluorine, it was brought into contact with a number of bodies whose density is known, comparing their behaviour at the same time in liquid oxygen, which has about the same boiling-point and density. By taking groups of bodies whose densities are very close to each other, it is easy to see which sink and which float in the liquid. This well-known though indirect method was the most suitable for these delicate experiments. The authors first satisfied themselves that the fluorine had no action on the materials used. To effect this, a crystal of ammonium thiocyanate (density = 1.31) was placed in a glass tube surrounded with boiling liquid air to the bottom of the tube, a current of fluorine gas was introduced by means of a platinum jet. The fluorine was rapidly liquefied, and the ammonium thiocyanate was not attacked. The same experiment was repeated with a fragment of ebonite ($d = 1.15$), of caoutchouc ($d = 0.99$), of wood ($d = 0.96$), of amber ($d = 1.11$), and of methyl oxalate ($d = 1.15$). It is of importance, in the experiments just mentioned, that the various materials used should be first kept at a temperature of -190° for some little time before coming in contact with liquid fluorine.

In one of the experiments a piece of caoutchouc, having been insufficiently cooled, took fire on the surface of the liquid, and burnt completely away with a brilliant flame without leaving any residue of carbon. The piece of caoutchouc ran about the surface of the liquid like sodium on water, giving a very intense light.

The density experiment was carried out in the following manner:—In a glass tube closed at one end, and of which the lower part had been slightly drawn out, fragments of the five substances just mentioned were placed. The tube was then plunged to a third of its length into boiling liquid air. When it was all reduced to a temperature of about -190° the fluorine gas was carefully intro-

duced. This soon liquefied, and the wood and the caoutchouc floated easily on the surface of the pale yellow liquid. On the other hand, the methyl oxalate and ebonite remained at the bottom, while the amber rose and fell in the liquid, appearing to be of the same density. The apparatus was shaken several times, and the quantity of liquid fluorine increased, but the results were the same.

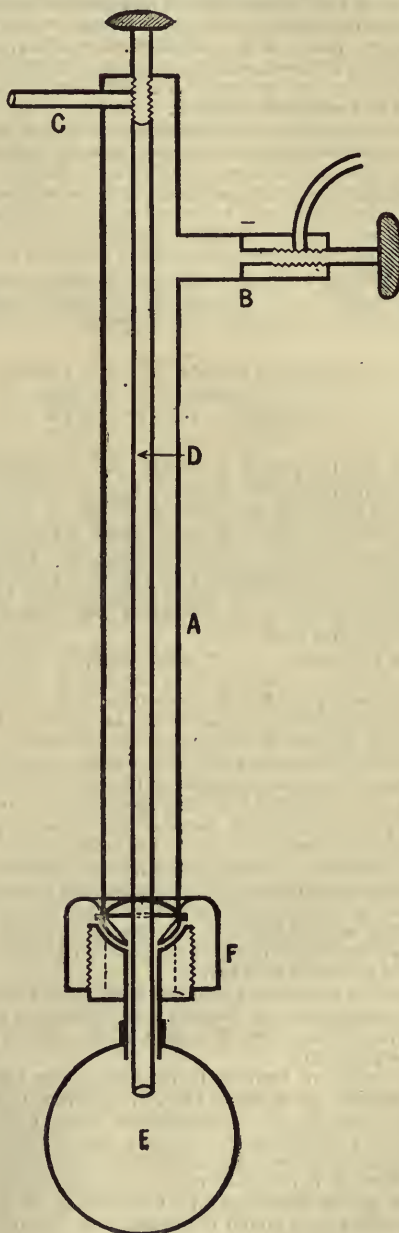


FIG. 1.

The authors thus arrive at the conclusion from these experiments that the density of liquid fluorine is about 1.14. Another point which appears to be of interest is the following:—The fragment of amber floating in the fluorine was very difficult to distinguish, which would seem to indicate that the index of refraction of liquid fluorine is in any case greater than that of liquid air or

oxygen, although it is not likely to be so high as that of amber itself.

Fluorine was liquefied in a thick-walled glass tube which had been previously graduated, and the tube sealed. On cooling the tube and its contents to -210° , a contraction of $\frac{1}{4}$ th in the volume of the liquid fluorine took place. A similar tube was left alone in a vacuum vessel full of liquid air. An hour and a half afterwards, the tube still being in liquid air, the fluorine had not changed in appearance. But shortly afterwards, when the air had all evaporated, a violent detonation occurred; the sealed tube and the double beaker in which it had been placed were smashed and reduced to powder.

Different samples of liquid fluorine examined with the spectroscope through a thickness of about $\frac{1}{4}$ c.m. showed no specific absorption-bands in the visible spectrum.

Liquid fluorine placed between the poles of a powerful electro-magnet does not show any magnetic phenomena. These experiments are the more decisive, as comparative ones with liquid oxygen were made at the same time.

The capillary constant of fluorine is smaller than that of liquid oxygen. A capillary tube, plunged successively in fluorine, oxygen, alcohol, and water, gave the following figures:—

Height of liquid fluorine	3.5 m.m.
" oxygen	5.0 "
" alcohol.. ..	14.0 "
" water	22.0 "

Liquid fluorine placed in a glass tube surrounded with liquid air (temperature about -190° C.) had a slow current of hydrogen gas directed on to its surface by means of a fine platinum jet. There was immediate combustion with the production of flame. The experiment was repeated by dipping the platinum jet well below the surface of the liquid. At this temperature complete combination still took place, with a considerable evolution of light and heat.

Oil of turpentine, in the solid state, is attacked by liquid fluorine. To perform this experiment a little oil of turpentine was placed at the bottom of a glass tube surrounded with boiling liquid air. As soon as a small quantity of fluorine was liquefied on the surface of the solid, combination took place with explosive force, a brilliant flash of light, and deposition of carbon. After each explosion the current of fluorine gas was kept up slowly, a fresh quantity of liquid fluorine was formed, and the detonations succeeded each other at intervals of from six to seven minutes. Finally, after a longer interval of about nine minutes, the quantity of fluorine formed was sufficient to cause, at the moment of the reaction, the complete destruction of the apparatus. In several of these experiments a little liquid fluorine accidentally fell on the floor; the wood instantly took fire.

The action of liquid oxygen has been studied with more care, since the authors observed that by passing a current of fluorine through liquid oxygen a detonating powder could be produced.

If a current of fluorine is directed to the surface of liquid oxygen in a glass tube, the temperature being about -190° , the fluorine dissolves in all proportions, imparting a yellowish colour, and giving the liquid a graded tint from the upper to the lower part; the bottom of the tube is hardly coloured. If, on the contrary, the fluorine gas is introduced at the bottom of the liquid oxygen, the yellow colour is produced at the bottom and diffuses slowly to the upper layers.

This phenomenon indicates that the densities of liquid fluorine and oxygen are very near each other. When the temperature of the mixture of liquid oxygen and fluorine is allowed to rise slowly, the oxygen evaporates first. The liquid becomes more and more concentrated as regards fluorine, and finally the latter begins to boil in its turn. In fact, at the commencement of this boiling the gas coming off will light a match which has only a red-hot point, and will not make lamp-black or silicon red-hot;

but, on the other hand, the gas coming off at the end of the experiment will instantly cause these two latter bodies to burst into flame. When the glass bulb is completely empty and its temperature is rising, a distinct disengagement of heat is suddenly noticed, and the interior of the glass loses its polish. This rise in temperature is due to the fluorine gas attacking the glass. In this experiment, using perfectly dry oxygen, no precipitate is produced. If, on the contrary, oxygen is used which has been some hours in contact with the air, the detonating substance mentioned in previous experiments is produced.

The body which is produced by the action of fluorine on oxygen containing in suspension minute crystals of ice seems to be a hydrate of fluorine, decomposing, with detonation, by a simple rise of temperature. This view must, however, be taken as conjecture, until the real composition is ascertained. A small quantity of water at the bottom of a glass tube being cooled down to -190° , liquid fluorine formed on the surface of the ice as a mobile liquid without showing any chemical action, and evaporated on the temperature rising. As soon as the apparatus became warmer the remaining gaseous fluorine attacked the ice with great energy, causing a strong smell of ozone.

A globule of mercury was treated in the same way as the water described above. The surface remaining very brilliant, the liquid fluorine surrounded it without causing any diminution of metallic lustre. On allowing the temperature to rise, the fluorine began to boil, and the liquid disappeared completely, without any attack of the mercury. The experiments seem to warrant the following conclusions.

Fluorine gas is easily liquefied at the temperature of boiling atmospheric air. The boiling-point of liquid fluorine is -187° . It is soluble in all proportions in liquid oxygen and in liquid air. It does not solidify at -210° . Its density is 1.14, its capillarity is less than that of liquid oxygen; it has no absorption spectrum, and it is not magnetic.

Finally, at -190° it has no action on dry oxygen, water, or mercury, but it reacts, with incandescence, on hydrogen and oil of turpentine. Future experiments must decide whether cooling below -200° can suspend the powerful chemical action of liquid fluorine on hydrogen and hydrocarbons.

One of the most important questions for future investigation is the specific refractive and dispersive indices of the fluid. Davy, in his paper on the substances produced in different chemical processes on fluor-spar (*Phil. Trans.*, 1813, 278), says, "Dr. Wollaston has found that the fluorine combinations have very low powers of refracting light, and particularly the pure fluoric acid; so that the refracting powers of fluorine will probably be found lower than those of any other substance, and it appears to possess higher acidifying and saturating powers than either oxygen or chlorine."

Gladstone has shown that the specific atomic refraction of the combined element does not exceed 0.9, taking the Lorentz formula, and that the atomic dispersion diminishes instead of increasing for short wave-lengths. Further, he found that the other halogen substitution compounds gave atomic refractions nearly agreeing with the same substances in the free state. It has been found that liquid gases give the same atomic refraction as the gaseous body, so that the refractive index of liquid fluorine may be at once deduced provided it behaves like chlorine, bromine, or iodine. Taking 0.9 as the atomic refraction, the value would be, according to the Gladstone formula, 1.054, and the Lorentz 1.081. Both values are far lower than those of liquid oxygen or air, 1.226 and 1.205 respectively. The general appearance of the liquid and the experiment with amber described above lead to the conclusion that liquid fluorine must have a refractive index much higher than that calculated. If the refractive index is as great as 1.41, then the atomic refraction (Lorentz) will be 4.13; but if it is only about 1.192, then the atomic

refraction will be 2. On both assumptions the atomic refraction of liquid fluorine is much greater than the value 0.9 found by Gladstone. Should the smaller value 2 turn out to be the correct one, then the inference might be fairly drawn that the critical constant was also about 3, or nearly the value for oxygen. This view would make the critical pressure of fluorine about the same as that of oxygen, or 50 atmospheres. From this it would follow that, unlike chlorine, bromine, and iodine, which have the same atomic refraction in combination and in the free state, fluorine has a different value in the one state as compared to the other. In this respect it would appear to resemble oxygen, whose atomic refraction in combination may be only three-fourths of what it is in the free state. This view is confirmed by an examination of the atomic volume of fluorine. The other members of the halogen series have approximately the same atomic volume in combination as in the free state. Now, the atomic volume of fluorine in fluorobenzene is 11.5, or about half the atomic volume of chlorine, or taking chlorobenzene as standard, with chlorine as 22.7, then the atomic volume would be 10. The value for the free element appears to be 16.6, and the number deduced from liquid hydrofluoric acid about 15. Many metallic fluorides have relatively small atomic volumes. Thus the fluorides of cadmium, lithium, calcium, magnesium, and aluminium have an atomic volume just about half of that of the corresponding chloride. This difference is, however, easily explained if fluorine in the combined state has only half the atomic volume of chlorine. Dr. Thorpe's value for the atomic volume of fluorine, deduced from a study of the chloride and fluoride of arsenic, is 9.2, or free fluorine at its boiling-point ought to have a density of 2, provided it behaved like the other halogens. This density for the free element is much too high, the experimental value being about 1.14. Such changes in atomic volume again suggest a resemblance with oxygen, and would lead to the inference that the refractive constants must also differ in the free and combined states. These interesting problems must, however, be left for future investigation.

(To be continued).

EDINBURGH UNIVERSITY CHEMICAL SOCIETY.

Monday, November 15th, 1897.

THIS meeting was held in the Chemistry Tutorial Classroom.

The President, Professor CRUM BROWN, delivered an address on "*The Chemical Work of Pasteur.*"

The President gave a short account of Pasteur's life, with an outline of his chemical and biological work, treating with more detail the discovery of left tartaric acid, and of the relation between crystalline and optical enantiomorphism.

With reference to Pasteur's views as to the connection between life and asymmetry, he said, "We often hear surprise expressed that Pasteur should have continued to hold that asymmetric molecules cannot be produced without the aid of living organisms, after he had himself shown that Perkin and Duppa's acid is racemic acid capable of yielding right and left tartaric acid. A careful study of what Pasteur actually said and wrote shows that this surprise is founded on a misunderstanding. Pasteur certainly believed at the time when he delivered his lectures to the Chemical Society of Paris (1860) that not only asymmetric substances, such as tartaric acid or glucose, but also racemoids, such as racemic acid, required the active presence of living things for their production. But in the note in the *Annales de Chimie et de Physique* in which he announces his discovery that Perkin and Duppa's acid is racemic acid, we see that he had already modified his view, and allowed that racemoids could be

produced by purely laboratory processes. And in 1874 and 1875, in communications to the Academy, he states his mature opinion in perfectly clear language. We may or may not agree with him, but his notions on the subject are not in contradiction to any observed fact."

As to the origin of the asymmetry in living things—as shown, for instance, in the fermentation of right but not of left tartaric acid by *Penicillium glaucum*—the speaker suggested that there may be some analogy between this and the development of asymmetrical habits in ourselves, such as the habit of following a definite order in putting on our stockings, shoes, &c.

On concluding his address Prof. Crum Brown was, on the motion of Dr. Macdonald, awarded a hearty vote of thanks for his very interesting address.

CORRESPONDENCE.

REFORM OF CHEMICAL AND PHYSICAL CALCULATIONS.

IN the CHEMICAL NEWS for 6th of August last (vol. lxxvi., p. 70) we published a review of a book having the above title. Mr. C. J. T. Hanssen, the author of the book, objected to some expressions used in the review, considering we had held him up to ridicule. The editor at once wrote to Mr. Hanssen assuring him that no such impression was intended, nor could it be fairly gathered from the tone of the review, which on the whole was distinctly favourable to the work. Our explanation not being considered satisfactory, and wishing to be scrupulously accurate in our statements and just to the author of the book, we think it will be better to let Mr. Hanssen speak for himself in the following letter, which we publish without further comment.

MATT., vii., 2.—"With what judgment ye judge, ye shall be judged."

3 Valdemarsgade,
Copenhagen, V., 20th November, 1897.

Professor Sir William Crookes, F.R.S.,
7, Kensington Park Gardens,
London, W.

DEAR SIR,

I received your esteemed and friendly letter of the 13th inst., and day after day I have read it and compared it with your review of my book in the CHEMICAL NEWS, but I regret to say I cannot in the review discover that you recognise the merit or any merit in my book. The first 35 lines are merely quotations from the book; then follows your judgment: "weird and forbidding aspect" of vulgar fractions; "a certain amount of fascination" in author's idea, but impossible to adopt it, gradual or universal; it would only increase the existing confusion!

A more sweeping condemnation of my book I can hardly conceive; and such a judgment, from a first-rate scientific authority, of course kills my book, and me as an author on scientific objects, unless you—in a distinct and prominent form, with your own signature—publish another review of my book in the CHEMICAL NEWS. Your friendly private letter, and the prefix Dr., which I am not entitled to, cannot mend the harm and damage done by the public condemnation.

If you had known my book, which, to judge from your letter, you do not, I am sure you would have written the review in a different style; but as matters now stand, I expect of you, as a gentleman, the satisfaction mentioned.

If you kindly will refer to the book, you will find that I by no means wish to do away with decimals, but freely use them myself where an approximately exact result is sufficient, but that I use vulgar fractions in the deter-

mination of *exact standard values*, which only in few cases can be found by decimals and by logarithms.

It was my aim to calculate standard values *exact*; approximate values, with unreasonable long tails of decimals, we have plenty of in the text-books of all nations, and they are the cause of the confusion which I wish to reform.

If we take Lord Rayleigh's determination of the weight of 1 cbm. of oxygen (latitude of London) = 1.42952 kg., the weight of hydrogen = exactly $\frac{1}{16}$ thereof, nitrogen = 14, carbon = 12 times the weight of H, then—

1 cbm.	Kg.	Kg.	Cbm.
H weighs	= 0.089345 = 1, and 1 is	= 1/0.089345 = 11.12945	
O	" = 0.42952 = 16, "	" = 1/1.42952 = 0.69953	
N	" = 1.250830 = 14, "	" = 1/1.25083 = 0.79496	
C	" = 1.072140 = 12, "	" = 1/1.07214 = 0.92745	
Argon	" = 1.786900 = 20, "	" = 1/1.78690 = 0.55647	
Helium	0.357380 = 4, "	" = 1/0.35738 = 2.78236	

These are calculated from Lord Rayleigh's standard for oxygen, as exact as can be done by decimals, but they are only approximate. Volume $\times$ weight is *not* = 1, as it should be.

In my system the conversion of weight to volume and *vice versa* is done without calculation, and the relation between both is always correct. The standard values (for latitude $41^{\circ} 10'$) corresponding to those above are expressed thus (page 3 § 9):—

1 cbm.	Kg.	And 1 kg.—	Cbm.	Cbm.
H weighs	5/56 = 1, H	= 56/5 =	11.2	
O	" 80/56 = 16, O	= 56/10 = 7/10 = 0.7		
N	" 70/56 = 14, N	= 56/70 = 4/5 = 0.8		
C	" 60/56 = 12, C	= 56/60 = 14/15 = 0.9333		
Argon	" 100/56 = 20, Argon	= 56/100 = 14/25 = 0.56		
Helium	" 20/56 = 4, Helium	= 56/20 = 14/5 = 2.8		

In the Dowson gas calculations, quoted with so much emphasis, you have overlooked that—

$$240 \times 867/140 \text{ is } = \frac{12 \times 867}{7}$$

(not so dreadful after all), and that this line belongs to the calculation of calors. per cbm.; and anyone looking over the next line may see at a glance that—

$$64/185 \times 4335 \text{ is } = \frac{64 \times 867}{37};$$

another very plain calculation. From § 25 the calors. per cbm. and per kg. may further be found by a very plain method, absolute exact.

In Table XXIII. the calors per cbm. and per kg. given for various gases. They are found, per kg., by multiplying 4335 cal. $\times$ ratios in col. 2; and per cbm. by multiplying 6192 $\frac{1}{2}$ = 43350/7 calors. $\times$ ratios in col. 6. Where, for mixed gases, the ratios are given in decimals, the results vary a trifle from the exact figures in cols. 1 and 5; the ratios in vulgar fractions for simple gases and chemical compounds give the exact values.

I think the chapters on evaporation and combustion are well worth your attention.

As the scientific world has adopted such complicated systems as the "Natural," the M.G.S., and the F.G.S., I think my plain arithmetic must be a release from the toil with Dynes, 1/746 h.-p. = 10^7 erg., &c., &c. After swallowing the camel, they cannot be afraid of the gnat.

I hope soon to be informed that this matter will be settled in a friendly manner, and remain

Very truly yours,

C. J. T. HANSEN, C.E.

NOTE.—*Erratum*: Table XXIII., col. 7, 3 lines from bottom, instead of 0.171 please read 0.240.

MOLECULES AND LIQUEFACTION HEATS.

To the Editor of the Chemical News.

SIR,—After reading the interesting paper by Noel Deerr (CHEMICAL NEWS, vol. lxxvi., p. 234) I cannot refrain from sending you an account of a thermal relation which I discovered a year or two ago, and which shows no less constancy than any of those which he refers to, while it has the important characteristic of being grounded on theoretical considerations. In fact, it was an *a priori* consideration of theory that led me to search for and to discover the relation.

Assume, then, in accordance with van 't Hoff's theory of solutions, that the molecules of a liquid have the same translational kinetic energy as they would have if they belonged to a gas of the same temperature. The specific heat of a liquid will consist of three parts:—Firstly, the heat required to increase the kinetic translational energy of the individual molecules; secondly, the heat required to increase the internal energy of the molecules, which in the case of monatomic molecules is nil; and thirdly, the heat required to overcome intermolecular attraction.

The first of these will be identical with the gaseous specific heat at constant volume. Now, when the liquid is changed into a solid the energy arising from this cause no longer exists in the translational condition, since the molecule becomes fixed in position; that energy will therefore be liberated as heat. The energy arising from the third cause possibly does not undergo any great change in amount, unless in solidifying the volume of the liquid greatly changes. The energy due to the second cause I neglect, in order to deal with monatomic molecules alone.

If these assumptions are sufficiently correct, it must follow that the heat set free in passing from the liquid to the solid state will, in the case of a monatomic molecule, be equal to the translational kinetic energy of a gas molecule at that temperature. Thus—

The molecular liquefaction-heat of a monatomic liquid is equal to the total kinetic energy of a monatomic gas molecule at the same temperature.

This for each grm.-molecule can easily be calculated, as is well known. Taking t the absolute temperature, it is equal to about $3t$ calories.

Doubtless the molecules in the liquid state are to some extent polymerised. Raoult's investigations on solvents seem to indicate that the ratio of the theoretical number of grm.-molecules is to the true number as 1:0.9 or as 1.1:1, this having a rough generality.

Hence the law just laid down with regard to liquefaction heats will be incorrect to this extent (if we neglect the internal changes in the heat of the few polymerised molecules). Since it is true, by a rough generalisation, that the theoretical number of molecules in the liquid is to the real number as 1.1:1 to 1, it will follow that the gaseous molecular kinetic energy calculated at the temperature of liquefaction will be greater than the true heat of liquefaction by the same ratio.

But since Raoult's number was derived from liquids remote from their solidifying-point, we expect greater polymerisation at that point, and therefore a somewhat larger ratio. We need not expect absolute uniformity between different liquids, but a ratio less than unity is inexplicable on this theory, except on the assumption of a great internal heat in molecules polymerised.

To show that these points are satisfactorily borne out, I give a table which I calculated some time ago, with the addition of six elements whose liquefaction heat was at that time unknown to me; I now take that constant from Deerr's paper.

The column headed M gives the molecular weight of the liquid (O = 16). We are here confined to liquid *metals*, which are the only elements that have any probability of possessing a monatomic molecule in the gaseous (or liquid) state. M.P. is the melting-point in absolute

degrees; L, the heat of liquefaction per unit mass; K, the gaseous kinetic energy calculated per unit mass at the same temperature. The last column is the ratio between these two.

	M.	M.-P.	L.	K.
Silver, Ag = 107.9	1227°	26.7	33.72	1.27
Cadmium, Cd = 112.1	588°	13.66	15.52	1.14
Mercury, Hg = 200.4	233°	2.82	3.45	1.22
Zinc, Zn = 65.5	685°	28.1	31.01	1.10
Platinum, Pt = 194.8	2048°	27.2	31.24	1.15
Lead, Pb = 206.9	605°	5.37	8.69	1.60
Palladium, Pd = 106	1773°	36.2	49.6	1.37

The following are after Deerr's numbers:—

Sodium, Na = 23	365°	32.7	47.48	1.45
Potassium, K = 39	335°	15.7	25.6	1.63
Copper, Cu = 63.3	1470°	43.0	55.3	1.29
Thallium, Tl = 204	560°	5.1	8.18	1.58
Aluminium, Al = 27	1150°	100.0	127.4	1.27
Gold, Au = 197	1330°	16.3	20.2	1.24

The following, which I calculated before, I found not to fulfil the law:—

Tin, Sn = 118.1	503°	13.62	12.63	0.93
Bismuth, Bi = 208	533°	12.64	7.6	0.603
Gallium, Ga = 69.9	303°	19.11	12.85	0.673

The first two have non-metallic tendencies, and their non-metallic relatives are allotropic.

On seeing Deerr's constants, I proceeded at once to make use of them in the calculation, and in no case was I disappointed. The result was six new metals well within the law. On the other hand, if we apply the principle to liquids of known polyatomic vapour or gas, we meet at once, as might be expected from theory, with enormous and disproportionate divergencies; this is in itself a confirmation. For example:—

	M.	M.-P.	L.	K.
Bromine, Br = 79.9	266°	16.2	4.98	0.303
Iodine, I = 126.9	386°	11.7	4.56	0.390

Higher molecules show still greater divergence. Still, it seems likely that an examination of such molecules (particularly those of similar structure) in the light of this theory would be fraught with important information respecting internal specific heats; and, indeed, information of a yet more far-reaching character.

I observe that Deerr mentions Crompton as giving a formula for a constant which involves atomic weight; probably it is similar to the above, or derived from it.—I am, &c.

P. J. BEVERIDGE, M.A., B.Sc.

St. Ann's, St. Helens, Nov. 15, 1897.

THE LATE VICTOR MEYER.

To the Editor of the Chemical News.

SIR,—There appears to be a strong desire among many of the British students who worked under the late Prof. Victor Meyer to give expression to the feelings of gratitude and admiration with which they remember him, by raising some form of memorial to be placed in the Heidelberg Lecture Theatre.

It has therefore been decided to call a general meeting of Prof. Meyer's British students, to be held in Manchester, on Saturday, December 11th, at 5 p.m. Prof. H. B. Dixon, F.R.S., has kindly placed the Organic Lecture Theatre of the Owens College at our disposal.

All past students of the late Victor Meyer, whether they worked with him in Zürich, Göttingen, or Heidelberg, are earnestly requested to be present.

I shall be pleased to receive suggestions from any who may be unable to attend, in order that they may be laid before the meeting.—I am, &c.,

J. J. SUDBOROUGH.

University College, Nottingham,
November 23, 1897.

ESTIMATION OF NICKEL IN STEEL.

To the Editor of the Chemical News.

SIR,—In the CHEMICAL NEWS (lxxvi., p. 248) you publish an electrolytic method of estimating nickel in nickel steel. I do not think that any electrolytic method is likely to supersede the xanthate process of Messrs. Andrews and Campbell, described in the Abstracts *J. C. S.* (Part II., 1895, p. 421).

I have used it many times, always with good results.

No figures appear to be given in support of Mr. Durn's method, where the percentage of nickel is very low.—I am, &c.,

H. L. ROBINSON.

Chemical Laboratory, Vickers, Sons, and Maxim,
Erith, Kent, Nov. 23, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 19, November 8, 1897.

Interpretation applicable to Faraday's Phenomena and Zeemann's Phenomena.—Henri Becquerel.

A Study of the Oysters of Cete as regards Pathogenic Microbia.—A. Sabatier, A. Ducamp, and J. M. Petit.—A pathological memoir.

Variation of Energy in Isothermic Transformations. On Electric Energy.—H. Pellet.

Dissemination of the X Rays.—A. Buguet.—The use of protective screens, which is not indefensible for short exposures before tubes of little penetrating power, becomes necessary in prolonged exposures. In medical applications they also render it possible to obtain more detailed proofs by shorter exposures.

On the Molecular Volumes and the Densities of Gases in general at Any Temperature and at Mean Pressures.—A. Leduc.—A mainly mathematical paper.

On some Novel Spectral Lines of Oxygen and Thallium.—H. Wilde.—On a comparison of the wavelengths of the lines it is found that none of them coincide with those of argon. Observations on the new lines of thallium 6955, 6560, gives the author occasion to again call attention to the spectroscopic study of thallium by Stas, in which he declared that the electric spectrum of this metal consists of a simple green line incapable of being split up like the spectrum of the flame. As the eminent Belgian chemist used in this research a current derived from a series of only 30 Bunsen elements the red line in the arc spectrum escaped his observation. To resolve the second line it is necessary for the current to have the intensity of 100 volts or 50 Bunsen elements. The red line 6560 in the arc-spectrum has been observed in all specimens of thallium and its compounds which have been submitted to examination.

The Use of Fluoresceine for the Detection of Traces of Bromine in a Saline Mixture.—H. Baubigny.—The author, in concert with P. Rivals, has devised a procedure for the decomposition of the bromides, founded on the action of a mixture of permanganate and a soluble salt of copper. He has sought for a practical means of deciding if this decomposition is complete at a given moment. The fluoresceine paper is obtained very easily. Fluoresceine is prepared by heating for three hours the desired proportions of orthophthalic acid, and of resorcin, to 190 to 200°; it is purified, and then treated with pure acetic acid at 40°—30° per cent. Into the filtered solution the paper is plunged, and allowed to dry. To use this paper it is moistened, when the least trace of bromine gives a distinct rose colour. Organic matter must be excluded.—*Comptes Rendus*, cxxv., No. 18.

MEETINGS FOR THE WEEK.

MONDAY, 29th.—Society of Arts, 8. (Cantor Lectures). "Gutta Percha," by Eugene F. A. Obach, Ph.D., F.C.S.
Royal Institution, 8.30. "The Wild Kafirs of the Hindu Kush," by Sir George Scott Robertson, D.C.L.

WEDNESDAY, Dec. 1st.—Society of Arts, 8. "The American Bicycle—The Theory and Practice of its Making," by Prof. Leonard Waldo, D.Sc.

THURSDAY, 2nd.—Chemical, 8. Ballot for the Election of Fellows.
"On Collie's Space-Formula for Benzene," by F. E. Matthews, Ph.D.

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of this Institute will be held on TUESDAY, the 11th day of January, 1898, and three following days.

In the event of it being found necessary to hold two Examinations, Candidates for the Final Examination will be examined from Tuesday, 4th, to Friday, 7th, of January, 1898.

Application forms and an outline of the Regulations can be obtained from the Secretary, at the above address.

Candidates are required to produce evidence of having passed a preliminary examination in subjects of general education and of having taken a systematic course of at least three years' study in one of the Colleges approved by the Council, or of having been engaged for two years in the laboratory of a Fellow of the Institute, and for two other years in one of the approved Colleges.

Full particulars are given in the book of Regulations for Admission to the Institute, which may be obtained from Messrs. Blundell, Taylor, & Co., 173, Upper Thames Street, London, E.C., price One Shilling.

By order of the Council,
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Candidates must apply for admission during the course of the preceding Term.

Forms of application can be had from the ASSISTANT SECRETARY, Royal Institution, Albemarle Street, W.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1984.

RESULTS OF EXPERIMENTS WITH THE CHEAVIN FILTER.

By Dr. T. L. PHIPSON,
formerly of the University of Brussels and the Laboratoire de Chimie
Pratique, Paris.

THIS cylindrical filter is made of porcelain (unglazed biscuit) like that known as the Pasteur-Chamberland, of which it is a modification, and in some respects an improvement. The object of this filter is to remove bacteria from water used for drinking; and it is applicable to other liquids for the same purpose.

It has been known for some time that these porcelain biscuit filters, when properly made and devoid of flaws, will remove microbes of all kinds from water which passes through them, and this has been proved in the present case by a number of experiments carried on in my laboratory during the summer months of the present year. But the passage of water through such a medium is, of course, very slow.

The yield may be increased, however, in three manners:—

1. By pressure; exerted, for instance, by a large cistern at the top of the house upon the filter in the basement.
2. By employing, with or without extra pressure, a certain number of cylinders instead of only one.
3. By producing a partial vacuum in the filter by means of a small hand pump, which pulls the water through the filter into the receiving vessel.

In the first case the cylinder is connected with a branch of the supply pipe at the basement; a single tube will thus yield each day sufficient water for drinking to supply a small family. When a larger supply is required, a certain number of the filters are joined together in a special cylindrical receptacle, and all act together.

In the third case, the use of a small hand pump, worked evenly and not too vigorously, will, by producing a partial vacuum in the receiving vessel and the porcelain cylinder, give a much more rapid yield of water also devoid of microbes. When the porcelain cylinder is placed thus in a vessel of stagnant water swarming with the "green matter of Priestley," consisting of extremely minute unicellular algae and various kinds of microbes, a few ounces may be pulled through in a few minutes, and the filtrate thus produced may be exposed to sunlight for weeks without the slightest green matter appearing, whilst ordinary river water exposed by its side as a "witness" becomes quite green.

In other experiments the water was filtered by means of the little hand pump into sterilised beef-tea and other media, with similar results.

With wines, spirits, beer, vinegar, solutions of sugar, solutions of salts, &c., the effects of the Cheavin filter are well worth attention. Cloudy wine is rendered bright without affecting its other properties. Cider, beer, and even oils can be thus rendered pure in a very short time.

When slime or mucilage has collected on the surface of the cylinders, they can be cleaned by brushing in boiling water, or by passing each tube slowly through a large naked flame. Being light and delicate in construction, these filters require some care in handling to avoid injury. At the old Fulham Potteries, where they are made in large quantities, a considerable number are broken up as unfit for use after testing. They are equally efficient for filtering air and depriving it of the microbes in suspension.

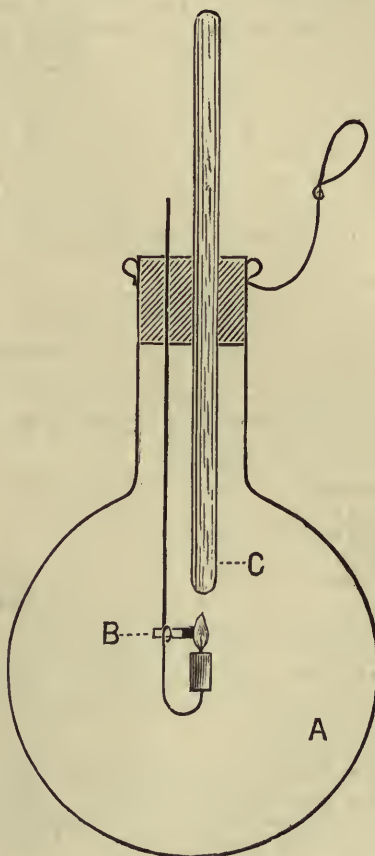
Casa Mia, Putney, S.W.

LECTURE EXPERIMENT.

By W. FRENCH.

I HAVE found the following experiment showing the effect of burning a candle in air to work extremely well, and it possesses the advantage of being very simple. It is a modification of an experiment well known, and figured in "Perkin and Lean," and dispenses with the use of a battery, which in a small laboratory is not always handy.

The round bottom flask, A (about 1 litre), is fitted with an indiarubber stopper through which passes an iron rod having a small candle attached. Just above the candle is



wired a small "match end," B, so that the head nearly touches the wick of the candle. C is a greased glass rod passing easily through the stopper. The indiarubber stopper can be wired down. The whole apparatus is counterpoised. The lower portion of the glass rod is heated, the cork replaced and wired in; the glass rod is now momentarily pushed down until it touches the match, which readily ignites the candle. When the candle ceases to burn re-weigh.

Obituary.—On November 5th C. W. Blomstrand, Professor of Mineralogy and Inorganic Chemistry at the University of Lund, closed his laborious and successful career in his 71st year.—*Chemiker Zeitung*.

Appointment.—Dr. F. Stanley Kipping, F.R.S., Lecturer and Assistant in the Chemical Research Laboratory of the Central Technical College, has been appointed to the Professorship of Chemistry in University College, Nottingham.

RESEARCHES ON SALINE SOLUTIONS.
LITHIUM CHLORIDE.

By GEORGE LEMOINE.

I HAVE sought to bring to the problem of the constitution of saline solutions new data, by the study of certain solutions which seem to present a peculiar interest.

Lithium chloride has the advantage of being exceedingly soluble; it is very stable and its molecular weight is very small ($\text{Li} = 7$). It is soluble not only in water, but in the ethylic, methylic, &c., alcohols. For these different solutions I have determined the heat of dilution, the density, and the solubility and the heat of solution, where it is not known. The calorimetric measurements have been effected by the methods of Berthelot. The proportions of the salt indicated result from gravimetric determinations of chlorine made as silver chloride. The lithium chloride employed had been previously purified with alcohol.

Lithium Chloride and Water.—We know a hydrate, $\text{LiCl} \cdot 2\text{H}_2\text{O}$ (Troost). The anhydrous salt, when dissolving in an excess of water, evolves 8.4 cals. per LiCl (Thomson). Its solubility has been determined by Kremers. We can prepare solutions containing up to about 13 LiCl per litre. I have determined the specific gravities at 0°.

Wt. of salt for 100						
grms. of the sol.	4'26	12'18	22'2	32'5	41'4	43'2
Sp. gr. at 0° ..	1'026	1'073	1'133	1'203	1'267	1'282

It is difficult to represent these experimental data by a curve without an inflexion like a parabola; they rather approach a group of two right lines. There seems then to be a modification in the constitution of the solution from about x_3LiCl to about 6LiCl per litre or from $\text{LiCl}_3\cdot\text{H}_2\text{O}$ to about $\text{LiCl}\cdot 8\text{H}_2\text{O}$.

The heats of dilution were measured at 10°.

Mols. LiCl per litre	12	9	6	3	1	0.5
Mols. H ₂ O for LiCl ₂ (cals.)	3.34	4.9	8.3	17.0	53.1	116
Quantities of heat of dilution starting from (12LiCl = lit)						
(cals.)	0	1.3	2.2	2.8	3.1	3.2

whence come the solution heats by admitting 8.4 cal. for excess of water, 5.2, 6.5, 7.4, 8.0, 8.3, 8.4 cal.

It seems that the solution heats increase regularly with the quantity of water. Beyond $\text{LiCl} \cdot 11\text{H}_2\text{O}$ there is no further disengagement of heat. Towards 20° the results were nearly the same.

Lithium Chloride and Methyl Alcohol (prepared from methyl oxalate and distilled over baryta). — M. Simon has described $2\text{LiCl} \cdot 3\text{CH}_4\text{O}$. According to my determinations the anhydride, when dissolving in an excess of alcohol, evolves 10.9 cals. for LiCl . I have measured the solubility and the specific gravity.

Temperature	1°	23°0'	50°
Ratio of the weight of the salt to the weight of the saturated solution..	0.26?	0.27	0.30
Weight of salt for 100 grms. of sol.	5.2	14.5	22.1
Specific gravity at 21.5°	0.836	0.910	0.974
" " at 0°	0.854	0.926	0.988

The dilution heats have been measured at about 18°; beyond (LiCl + 48CH₄O) there is no appreciable disengagement of heat.

Mols. LiCl per litre.. .. .	5	3	1	0.5
Mols. CH ₃ O per LiCl	4.7	7.9	24	48
Quantities of dilution heat starting from 5LiCl+1 litre (cals.)	0	1.5	2.6	3.0
Whence come the solution heats with 10.9 cals. for an excess of alcohol (cals.)	7.9	9.4	10.5	10.9

Lithium Chloride and Ethylic Alcohol (rendered anhydrous by distillation with baryta).—M. Simon has described $\text{LiCl} \cdot 2\text{C}_2\text{H}_5\text{O}$; my analyses give the same

formula. The anhydrous salt, on dissolving in an excess of alcohol, evolves 11.7 calories for LiCl (M. Pick). I have found for its solubility—

Temperature	16°	57°	13.0°	25.0°	40.6°	62.6°
Ratio of weight of the salt to the wt. of the saturated solution	0.14	0.14	0.13	0.14	0.15	0.18

The graphic representation corresponds approximately to two right lines which intersect each other about 30°, a very small angle; the more oblique line being inclined slowly towards the temperature of fusion (about 600°). The solubility decreases progressively from water to ethylic alcohol, and thence to amylc alcohol, in proportion as the molecular weight $C_n H_{2n+2}O$ increases.

Weight of salt in 100 grms.				
of solution.. ..	0	5.2	10.1	14.6
Specific gravity at 14°2'	0.797	0.839	0.871	0.903
" " at 0°.. ..	0.809	0.851	0.881	0.903

The dilution heats have been measured between 8° and 15°; beyond $\text{LiCl} + 35\text{C}_2\text{H}_6\text{O}$ no perceptible degree of heat is evolved.

Mols. LiCl per litre	3	2	1	0.5
Mols. C ₂ H ₆ O per LiCl.. ..	5.4	8.3	16.9	35
Quantities of dilution heat, starting from 3LiCl=1 litre (cals.)	0	8.3	2.1	2.6
Hence solution heats with 11.7 for an excess of alcohol (cals.)	9.1	8.3	11.2	11.5

—*Comptes Rendus*, cxxv., No. 17.

ON THE ESTIMATION OF PHOSPHORIC ACID.

By HENRI LASNE.

I.

SOME years ago I communicated to the French Chemical Society (*Bull. Soc. Chim.*, 1889) some experiments on the estimation of phosphoric acid. All that I have since done on this subject has confirmed my former results, which I will now briefly recal.

1. The precipitation of the ammonio-magnesium phosphate gives rise to no loss when carried out in the presence of citrate of ammonia and a sufficient excess of magnesia.

2. The lime, oxide of iron, alumina, and manganese are not carried down by the precipitate.

3. The presence of silica and of fluosilicates causes an excess.

4. Without the addition of an excess of magnesia, the precipitation is not complete, and the filtrate will give a precipitate either with magnesia or phosphoric acid.

These results have been recently confirmed, and they demonstrate that, starting with the ammonio-magnesian phosphate, this compound can be reproduced without any loss whatever; after calcining, we obtain exactly the same weight.

The question is—Has this calcined body the theoretical composition of the pyrophosphate? Nothing seems to prove it; since the sample under analysis was obtained under exactly the same conditions as the final precipitate. We shall see later how some doubts have arisen on this subject—doubts which I may say were without excuse. Because of the great importance of the exact estimation of phosphoric acid, and of the very great accuracy with which it can be carried out, a detailed account of my experiments on the subject may prove to be of use.

II.—On the Influence of Time of Standing.

It is well known that the precipitation of ammonio-magnesian phosphate requires a certain amount of time,

and it has been generally recognised that twelve hours at least was necessary. Some chemists have maintained that filtration could be proceeded with much sooner; I, in my experiments on this particular point, have met with some anomalies, of which the following is a good example:—

From a solution of a natural phosphate, freed from silica, I took nine equal volumes of 50 c.c. each (corresponding to $\frac{1}{2}$ grm. of the phosphate). To each of these samples I added, as usual, 25 c.c. of citrate of ammonia at 100 grms. of citric acid per litre, 60 c.c. of ammonia at 22°, and 20 c.c. of ammonio-magnesian chloride at 20 grms. of magnesia per litre.

The washings were effected with 45 c.c. of water containing one-third of its volume of ammonia at 22°, making a total volume of 200 c.c.

After agitation and the formation of the precipitate, the solutions were let stand for varying times. The results obtained are given in the following table:—

No.	Time of standing.	Weight of precipitate.	Remarks.
I.	1 hour	0.2329	Filtrate cloudy.
II.	2 hours	0.2338	Filtrate slightly cloudy.
III.	4 "	0.2350	Clear.
IV.	8 "	0.2351	"
V.	16 "	0.2345	"
VI.	32 "	0.2345	"
VII.	64 "	0.2342	"
VIII.	128 "	0.2338	Loss probable.
IX.	256 "	0.2343	"

It is seen at once that the weight of the calcined precipitate first of all increases for eight hours, then slowly declines to a definite limit in about sixteen hours (Experiment No. VIII. would appear to be vitiated by some accident). The above proves the occurrence of two superposed phenomena, which may be, for example:—

1. The precipitation of the phosphoric acid which takes place at once, more or less, in the state of tri-magnesian phosphate and is complete in about eight hours.

2. The much slower transformation of the tri-magnesian phosphate, at first formed as ammonio-magnesian phosphate, which takes at least sixteen hours.

These results have been confirmed by a large number of isolated experiments—which have, as a matter of fact, been the cause of the present systematic research. Two examples are here given:—

	I.	II.
Standing for 4 to 6 hours..	0.2176 grm.	0.2175 grm.
Standing for 16 to 22 hours	0.2163 "	0.2166 "

The excess in weight of the precipitate collected between four and six hours may therefore exceed that which has stood for sixteen to twenty-two hours by at least 1 m.grm. However, these facts being susceptible of different interpretations,—different, in fact, to that which has been given above,—it becomes necessary to look for a definite answer, and to primarily enquire into other experiments showing similar variations.

III.—Mechanical Precipitation.

It has been well known for some years, and even adopted officially in Belgium, that continuous shaking for a quarter of an hour or twenty minutes of the solution to which the precipitant has been added is of great advantage. This has been called the *citro-mechanical* method.

The comparative examination I have made of this method leads me to the conclusion that it gives exactly the same results, in weight of the calcined precipitate, as in letting stand for four to six hours; that is to say, an excess of about 1 m.grm. above the results obtained after sixteen to twenty hours. This conclusion has been arrived at from a large number of estimations carried on by the two methods simultaneously.

Is there really an excess in the case of rapid precipita-

tions, or does the long standing bring about a loss? What has been written up to now only shows that there is a decided difference between the two methods.

IV.—On the Influence of Dilution.

Two experiments were made with equal volumes of the same solution. The quantities of citrate and chloride of magnesium used were the same in each case; but in the second the total volume was doubled by the addition of water, but keeping the proportion of free ammonia constant. The following table gives the conditions and the results of the two experiments:—

	I.	II.
Solution of phosphate	50 c.c.	50 c.c.
Citrate of ammonia	25 "	25 "
Ammonia at 22°	60 "	120 "
Chloride of magnesia	20 "	20 "
Water	—	95 "
Wash-waters ($\frac{1}{2}$ ammonia at 22°)	40 "	80 "
Total volume	195 "	390 "

Weight of calcined precipitate.. 0.2336 grm. 0.2353 grm.

Contrary to what might be expected, the greater dilution causes an augmentation in the weight of the precipitate collected.

This systematic experiment was suggested by a certain number of results obtained in the ordinary course which had led to the same conclusions; the fact now appears to be beyond doubt.

Here, again, several explanations are possible. Either the nitrate of ammonia exercises a solvent action on the ammonio-magnesian phosphate,—an action which is the more marked as the solutions are the more concentrated,—or there remains in the precipitate tri-magnesian phosphate or phosphate of lime, proportionally greater as the solutions are more dilute.

V.—Does the Excess come from Lime?

One might suppose that in the first moments of precipitation lime, in the state of phosphate, would be carried down; its further transformation being only very gradually achieved. I was the more inclined to this belief, inasmuch as I had just come across a body which did not appear to have been previously noticed, the *ammonio calcic phosphate*.

This compound is obtained under exactly the same conditions as the ammonio-magnesian phosphate in the presence of citric acid in a strongly ammoniacal solution. It is fairly soluble in ammoniacal water, and is formed more easily at a low temperature. The precipitate is composed of small brilliant crystals, sufficiently large to be recognised by the naked eye. Its composition, according to an analysis I have made, is calculated absolutely on that of ammonio-magnesian phosphate. In spite of its relative solubility, it does not appear impossible, by reason of isomorphism, that this phosphate might be at first carried down with the ammonio-magnesian phosphate, and then slowly decomposed. I have searched—and searched in vain—to see if there is any trace of lime in the precipitates showing an excess; but there is none, and this result is beyond doubt; for to produce an excess of 1 m.grm. it would be necessary for 3 m.grms. of magnesia to be replaced by 4 m.grms. of lime—a quantity which could not fail to be detected. It is therefore necessary to put on one side the hypothesis of the presence of lime in ammonio-magnesian phosphate.

VI.

With the object of finding out the reason for these slight divergencies I carried out another series of experiments which I will now proceed to describe.

Three equal volumes of a similar solution, each corresponding to 1.25 grm. of the natural phosphate, were pre-

precipitated with proportional quantities of the usual reagents under the following conditions:—

- I. Let stand for twenty-one hours.
- II. Let stand for three hours and a half.
- III. Mechanical precipitation in twenty minutes.

After thorough washing, the three precipitates were dissolved in hydrochloric acid, and from each solution made up to 250 c.c. two quantities of 100 c.c. were taken, corresponding to 0.50 grm. of the original phosphate. Thus we get two series: the three solutions of Series *a* were precipitated with 10 c.c. of chloride of magnesium to estimate the phosphoric acid; and those of Series *b* with 10 c.c. of a solution of phosphate of ammonia at 10 per cent to estimate the magnesia. At the same time, 50 c.c. of the original solution were submitted to direct precipitation and gave 0.2507 grm. of calcined precipitate (after standing sixteen hours). The following are the results obtained from the two series, *a* and *b*, after standing for sixteen hours.

	<i>a.</i>	<i>b.</i>	Difference : <i>b</i> - <i>a</i> .
I.	0.2508 grm.	0.2700 grm.	0.0192 grm.
II.	0.2506 "	0.2738 "	0.0232 "
III.	0.2498 "	0.2727 "	0.0229 "

These results were most unexpected. It certainly appears as if the Series *a* corresponds with the usual method for the estimation of phosphoric acid, since the concordance between the direct estimation and the result (I., *a*) which corresponds to it are as close as possible; but II., and above all III., in spite of the excess always caused by rapid precipitation, contain less phosphoric acid. This excess would appear, therefore, to be due to an excess of magnesia; and this would be confirmed by the figures of column *b*, which we may consider as proportional to the quantities of magnesia in the original precipitates.

The considerable differences between the corresponding figures of columns *a* and *b*, which should be identical, or at least very close to each other, present an important question to be solved.

1. Either in the original precipitates, and in Series *a*, there is a large excess of magnesia. It would then be necessary that, in the method as usually practised, tri-magnesian phosphate be formed; the estimation of the phosphoric acid being in such a case tainted by a serious error.

2. Or, in Series *b*, there is an excess of phosphoric acid. The weighed precipitate would then contain metaphosphate mixed with pyrophosphate, and to explain this fact it is necessary to admit that bi-ammoniacal magnesian phosphate is precipitated at the same time as the normal ammoniaco-magnesian phosphate. The estimation of the magnesia would then be erroneous.

3. Or, further, that the two causes of error co-exist.

To decide which of these three hypotheses is correct, the preceding experiments are not sufficient, and it became necessary to have recourse to rigorous synthetical trials.

VII.—Trials with a Known Quantity of Phosphoric Acid.

It is not advisable to use ammonio-magnesian phosphate to obtain a known quantity of phosphoric acid; phosphate of ammonia is far preferable. But if we can make sure of the purity of this body, in so far that it contains nothing but phosphoric acid and ammonia, its hygroscopic state and its degree of basicity need not be rigorously defined. So as to start from an absolutely certain base, after having made a solution containing about 34 grms. per litre of pure phosphate of ammonia, I caused 10 c.c. of this solution to be absorbed by pure magnesia, strongly calcined, and after drying at 100° it was again calcined (the calcination should last about three-quarters of an hour). The difference between the two weightings

gives, for 10 c.c., 0.1801 of phosphoric acid, with possible variations of one-tenth of a m.grm., more or less. Other trials made with 20 and 30 c.c. gave proportional results, with the same limits of error. We can therefore feel sure of the titration value of the solution.

Samples of 10 c.c. each were precipitated under the ordinary conditions by the addition first of 40 c.c. of water, then of definite quantities of citrate of ammonia and chloride of magnesium. The average of these experiments gave a weight of pyrophosphate of 0.2823 grm., with variations not exceeding two-tenths of 1 m.grm., more or less. This corresponds to 0.1802 grm. of phosphoric acid. From this we conclude that the precipitation is complete, and that the pyrophosphate of magnesia is of normal composition under the afore-mentioned conditions; that is to say, after sixteen hours standing in a volume of 150 c.c. containing 10 grms. of citric acid and at least one-third of its volume of free ammonia, and, finally, an excess of magnesia.

I have repeated the experiment under the same conditions, but using hydrochlorate of ammonia instead of citrate. This seems permissible, since there is no other base but magnesium to keep in solution, but the results cannot be depended on, and there is always a large excess. The weight of the calcined precipitate is increased to 0.2976 grm., which corresponds to 0.1903 grm. of phosphoric acid, taking it to have the composition of the pyrophosphate. This precipitate thus contains such a large proportion of tri-magnesian phosphate that even standing for sixteen hours does not suffice to transform it into ammoniaco-magnesian phosphate. This observation is of importance, inasmuch as it is often recommended to precipitate phosphoric acid after the molybdic separation, for example, by a simple addition of hydrochlorate of ammonia and of magnesia. Results obtained in this manner are quite untrustworthy.

VIII.—Experiments with a Known Quantity of Magnesia.

A known quantity of strongly calcined magnesia was dissolved in hydrochloric acid, then precipitated in the presence of citrate of ammonia by an excess of phosphoric acid; the quantity of phosphoric acid in excess was varied by using increasing quantities of a solution of 100 grms. per litre of phosphate of ammonia (titrating 0.54 grm. of phosphoric acid per 10 c.c.).

Two series of experiments were made:—

A on 0.0948 grm. of magnesia.
B " 0.1034 " "

In each series three precipitations were made, with 5, 10, and 20 c.c. of the solution of ammoniac phosphate respectively. The following results were obtained:—

	<i>A.</i>	<i>B.</i>
Magnesia used	0.0948 grm.	0.1034 grm.
Corresponding pyrophosphate	0.2630 "	0.2868 "
Weight of calcined precipitate obtained with—		
5 c.c. ammoniac phosphate	0.2687 "	0.2929 "
10 " "	0.2709 "	0.2981 "
20 " "	0.2779 "	0.3107 "

We can see from the above that even with a slight excess of phosphoric acid (5 c.c. leaving not more than 0.1 of phosphoric acid in the solution) there is an excess—due, without doubt, to the mechanical carrying down of the phosphoric acid by the precipitate. This excess further increases rapidly as the solution is richer in phosphoric acid.

For the purpose of confirming this, a third series, C, was made under the same conditions as B; but instead of weighing the precipitates at once, they were dissolved for the purpose of estimating the phosphoric acid in the ordinary manner. The following results were obtained:—

	C.
Magnesia used.	0.1034 grm.
Corresponding phosphoric acid	0.1834 "
Phosphoric acid obtained in the precipitate by—	
5 c.c. ammoniac phosphate	0.1940 "
10 " " " " " " " "	0.1971 "
20 " " " " " " " "	0.2064 "

It is seen thus that the precipitate carries down a large excess of phosphoric acid, which, on calcination, gives metaphosphate.

These results confirm and explain those which have been already obtained, notably in Section V.

Although I have not been able to isolate a bi-ammoniacal magnesian phosphate, everything points to the existence and formation of this compound, the more abundantly as the reaction takes place in a greater excess of ammoniac phosphate.

IX.—Conclusions.

Finally, we deduce from what has been stated the following consequences:—

1. The estimation of phosphoric acid, in the state of pyrophosphate, without any precautions beyond the preliminary elimination of the silica, gives trustworthy results, untainted by systematic errors.
2. Rapid precipitations cause an excess, due to the partial formation of trimagnesian phosphate, which is only transformed into ammonio-magnesian phosphate after sixteen hours contact with sufficiently concentrated citrate of ammonia (10 grms. of citric acid to 150 c.c. of solution). It therefore follows that to obtain satisfactory results the solution should be allowed to stand all night.
3. In spite of all this, the excess is sufficiently small as not to completely condemn these rapid methods, which can with advantage be used commercially if due notice is given that this is being done.
4. The transformation of the trimagnesian phosphate into ammonio-magnesian phosphate is very slow in the presence of hydrochlorate of ammonia only, and it should always be remembered to add the necessary quantity of citrate.
5. The precipitation of the magnesia in the presence of an excess of ammoniacal phosphate gives, at the same time, with the ammonio-magnesian phosphate another phosphate, not only poorer in magnesia, but also poorer as the excess of phosphoric acid present is greater. The estimation of the magnesia by this almost classic method is, therefore, always erroneous. This is a subject to which we shall return later on.—*Bull. Soc. Chim.*, vols. xvii.-xviii., Nos. 16 and 17, 1897.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, November 4th, 1897.

(Continued from p. 263).

DISCUSSION.

DR. PERKIN said he felt much interested in the paper, because of the remarkable magnetic rotation of combined fluorine, for example, in fluorobenzene. When one atom of hydrogen in benzene is displaced by chlorine, the rotation is considerably increased. The substitution of bromine causes a still higher rotation, and that of iodine the highest. On the other hand, the substitution of fluorine reduces the magnetic rotation. He had suggested that this might be accounted for if fluorine were paramagnetic, because its magnetic rotation would then be the reverse of that of carbon and hydrogen. This, however, does not seem to be a probable explanation since it is now found that liquid fluorine is not paramagnetic. It

is possible that this element may have different values depending on whether it is free or combined. The nitro-group (NO_2) influences magnetic rotation much in the same way as fluorine.

Dr. GLADSTONE remarked on the importance of Prof. Dewar's communication, the most interesting portion to him being that on the optical properties of the liquid fluorine. The specific refraction of that element had been calculated by him and his brother from fluorobenzene and from many salts, crystallised or in solution, with the invariable result that it was exceedingly small. In the last list of the specific refractions of the elements (*Proc. R. S.*, 1897, lx., 141) it is given at only 0.031, which is not a third of the next lowest in the list. Its specific dispersion is also low, and it has the additional peculiarity of giving a reversed spectrum. Now Prof. Dewar finds that liquid fluorine has about the same refractive index as that of amber; this is known to be 1.55 or thereabouts. As the specific gravity of the fluorine is stated to have been 1.14, we can easily calculate the specific refraction, viz., 0.482. This figure, instead of being the lowest in the list of elements, is nearly the highest, there being only six with higher values.

It is true that in some cases the specific refraction of an element in the free state differs somewhat from that deduced from its compounds. Fluorine would naturally be compared with the three halogens, chlorine, bromine, and iodine. Liquid chlorine has a specific refraction of about 0.27; in combination 0.28. Bromine has a specific refraction of about 0.20; in combination 0.21. Iodine vapour has a specific refraction of about 0.19; in combination 0.21. The free element, therefore, does not differ widely in specific refraction from the same element when in combination, and in each case is the smaller and not the greater of the two. A certain analogy does exist between fluorine and sulphur or phosphorus. These two when melted have high specific refractions, sulphur being 0.50 and phosphorus 0.59; these high figures are generally much reduced when the elements are in combination, but the extent of this reduction is by no means comparable with what would appear to be the case with fluorine. Although Professor Dewar's method is correct in principle, Dr. Gladstone expressed a strong hope that accurate determinations would be made by one or other of the more direct methods.

Dr. THORPE said, in reference to the allusion by the President to his determination of the specific molecular volume of fluorine as far back as 1880, that too much stress could not be laid upon the particular value, viz., 9.2, which he then obtained. It was deduced from a study of the specific gravity and thermal expansion of arsenic fluoride, a substance which is not easy to obtain pure, and which is not altogether without action on glass, especially at temperatures approaching the boiling-point. It, moreover, presupposes that arsenic fluoride has a molecular constituent analogous to that of arsenic chloride. Such an assumption is probable; but having regard to the remarkable complexity of many fluorine compounds, as, for example, hydrogen fluoride itself, when compared with the corresponding chlorine compounds, the supposition cannot at present be regarded as more than probable. The particular value obtained, however, clearly indicated the order of the magnitude, as shown by its substantial agreement with the other values quoted by the author.

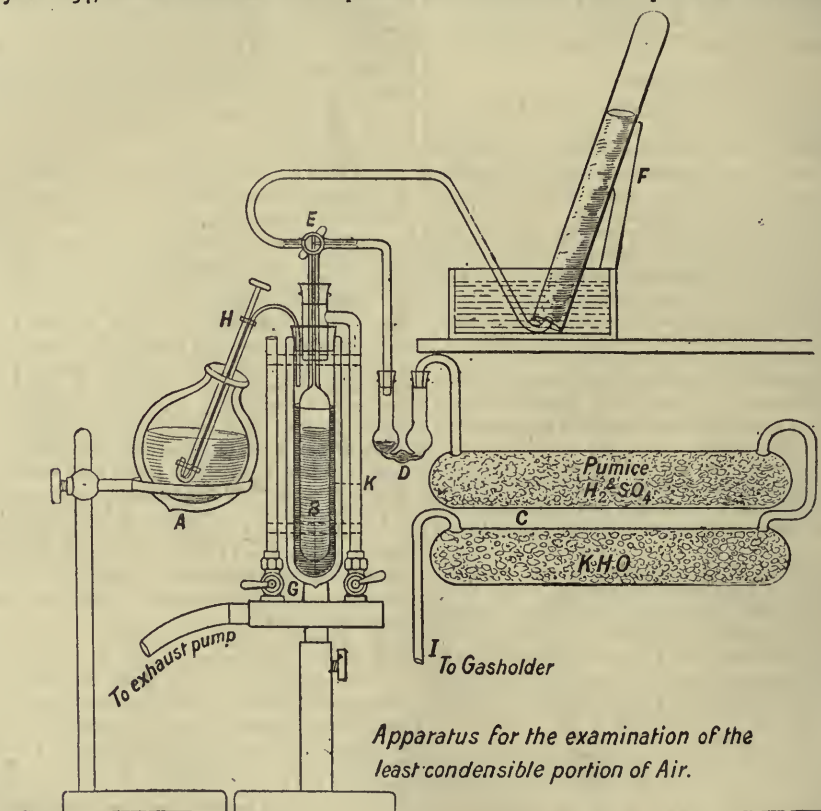
With respect to the question raised by Dr. Gladstone he might say that the peculiar behaviour of glass when immersed in arsenic fluoride was significant, and suggested a method by which the refractivity of liquid fluorine in the free state might be ascertained with a fair approximation to accuracy, viz., on the same principle as that adopted by the authors in determining the relative density of liquid fluorine—that is, by immersing solids of known refractivity in the liquid, and observing which became invisible. Arsenic fluoride is a highly refractive liquid, and some specimens of glass threads and tubes become almost invisible when immersed in it.

Professor DEWAR, in reply, observed that he did not intend to convey the impression that, because amber in liquid fluorine might be difficult to define clearly, it necessarily followed that the refractive index would turn out to reach 1.55, as Dr. Gladstone seemed to infer. His present impression was that it exceeded that of liquid air, but he could go no further. No doubt the next time Professor Moissan and he had the opportunity of continuing the experiment, a direct determination of the refractive index would be made.

*112. *The Liquefaction of Air and the Detection of Impurities.* By Professor DEWAR.

In a paper on "The Relative Behaviour of Chemically-prepared and of Atmospheric Nitrogen," read before the Society in the year 1894, it was stated that all samples of

holds the liquid air maintained under continuous exhaustion. As this low temperature had to be kept steady for from one to two hours, while at the same time the bulb B had to be completely covered with liquid air, it was necessary to arrange some means of keeping up the liquid air supply without disturbing the apparatus. The plan adopted is shown at H, which is a valve arrangement which can be so regulated as to suck liquid air from the large vacuum vessel A, and discharge it continuously along a pipe into the vacuum test-tube G, the latter being kept under good exhaustion. In working the apparatus the tube I is connected to a gasometer containing 10 cubic feet of air, so that the volume of air condensed in each experiment may be observed. This was generally from 2½ to 3 cubic feet. If there is a very small proportion of some substance not liquefiable or soluble in liquid air



Apparatus for the examination of the least condensable portion of Air.

FIG. 2.

nitrogen and oxygen properly purified, are, when liquefied, clear transparent liquids, so that the solid matter which always separates when air or nitrogen or oxygen is liquefied on the large scale consists of impurities. Ordinary air, containing 4 parts of carbonic acid per 10,000 parts, gave a turbid liquid from the solidification of the carbonic acid; and oxygen containing traces of chlorine behaved in a similar manner. With the object of ascertaining the proportion of any gas in air that is not condensable at about -210° C. under atmospheric pressure, or is not soluble in liquid air under the same conditions, the following apparatus has been devised:—A cylindrical bulb of a capacity of 101 c.c., marked B in figure, had a capillary tube sealed into it terminating in a three-way stopcock, as shown at E. The parts marked C and D consist of soda-lime and sulphuric acid tubes for removing carbonic acid and water. The stand marked G holds the large vacuum test-tube into which B is inserted which

then we should expect the vessel B would not fill up completely into the capillary tube. This is, however, exactly what does take place. After forty minutes' cooling, the vessel B and the cool part of the tube were filled with liquid. In this experiment some 80 litres of air were condensed, and any accumulated uncondensed matter must have been concentrated in the upper part of the capillary tube which had a volume of 0.5 c.c. Under the conditions, therefore, the material looked for must be less than 1 part by volume in 180,000 of air.

To test the working with an uncondensable gas added to air, a volume of 10 cubic feet was taken in the gas-holder, and to that 500 c.c. of hydrogen were added. This is in the proportion of less than 1 in 500. Even after two hours' cooling, the tube B could only be filled four-fifths. In order to prove that the gas accumulated in the upper part of B was hydrogen, the three-way stopcock at E was turned, and the temperature allowed to rise

so that the gas was expelled from the evaporation of the liquid air and collected over mercury as shown at F. The gas thus collected was easily combustible, and consisted chiefly of hydrogen. The amount of hydrogen was then reduced to 1 part in 1000 of air, and it was found that after one and a quarter hour's cooling the bulb B had filled to within a $\frac{1}{2}$ c.c. of the capillary tube. A new sample of air containing 1 part of hydrogen in 10,000 of air filled the bulb B completely as if it were ordinary air.

It appears from these experiments that 1 part of hydrogen in 1000 of air is just detectable by this plan of working. As the 80 litres of air condensed contained some 80 c.c. of hydrogen, it appears that 100 c.c. of liquid air at from -200° to -210° C. had dissolved nearly all this gas; in fact, that 20 c.c. of hydrogen at the low temperature is dissolved in 100 c.c. of liquid air. In the

precipitate by transmitted light looked yellow-brown. This solid turns out to be of organic origin, probably of the petroleum order of compounds. It has a very marked aromatic smell resembling such bodies. The trace of material left gave, after treatment with concentrated nitric acid, the smell of nitrobenzene; and as its detection cannot be explained by the presence of any material of the kind in the vessels used in collecting, it must be assumed to be a normal constituent of the Bath gas. A further quantity of the Bath gas must be collected in order to confirm the presence of such bodies and to definitely make out their nature. Another peculiarity of the liquid is that, on examining it with the spectroscope, even through a thickness of 2 inches, no trace of the characteristic oxygen absorption spectrum could be detected. In all attempts to make nitrogen for liquefaction on the large

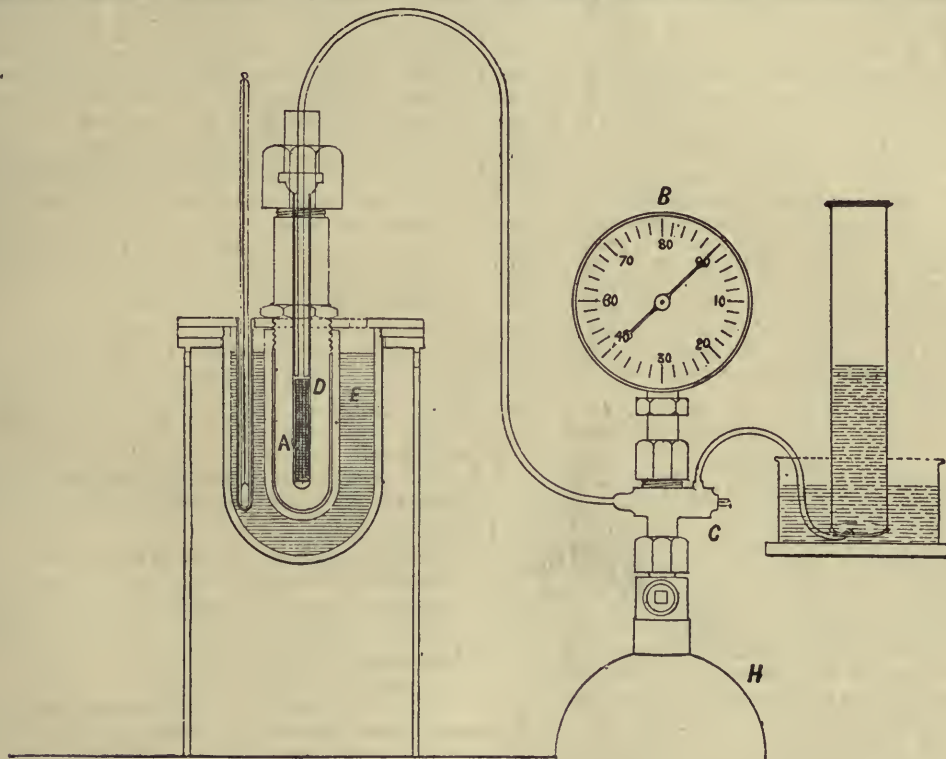


FIG. 3.

paper on "The Liquefaction of Air and Research at Low Temperatures" (*Proc.*, 1895, xi., 221) it was shown that if hydrogen containing a small percentage of oxygen were employed for the purpose of getting a hydrogen jet, the liquid collected from it was oxygen, containing, however, so much hydrogen dissolved in it that the gas coming off for a time was explosive.

In order to press this inquiry a little further, some natural gas known to contain a different constituent like helium suggested itself as being worthy of trial. Lord Rayleigh's results of the examination of the gas from the King's Well at Bath showed that it contained 1.2 part of helium per 1000 volumes, so that it seemed admirably adapted for such experiments. The author has to express his thanks to the Corporation of Bath for giving permission to collect samples of the gas.

The sample of gas from the Bath Spring was treated exactly in the same way as the hydrogen mixtures described above. During the liquefaction there was a marked difference in the appearance of the liquefied gas, for while the hydrogen and air mixtures gave a clear, transparent liquid, the product from the Bath gas was turbid, and the

scale, oxygen could always be detected in the liquid with the greatest ease by means of its absorption spectrum. After the cooling had continued for one hour the gas ceased to flow into the condensing vessel, and some 20 c.c. at the upper part of the glass cylinder B was filled with a gas that had not undergone liquefaction or solution. About 70 litres of the Bath gas were condensed, certainly the largest quantity of this gas ever subjected to chemical examination. This was boiled off just as the hydrogen was treated in the experiments described above, and as, by accident, too much nitrogen had volatilised along with the gas, oxygen was added and the mixture sparked over alkali to get rid of the excess of nitrogen. During the sparking the helium lines were well marked (along with others the origin of which must be settled later), and a vacuum tube filled with the product of the sparking gave a splendid spectrum of the gas. The sample of gas directly collected from the liquid nitrogen contained about 50 per cent of helium. It is therefore possible to separate helium from a gas when it is only present to the extent of one-thousandth part by liquefaction in the manner described. From this it would appear that helium is less

soluble in liquid nitrogen than hydrogen is in liquid air, and is of greater volatility than either of the constituents of air, as Professor Olszewski found (*Bull. Ac. Crac.*, 1896, 297) by direct experiment on a pure sample of the gas sent to Cracow by Professor Ramsay with the object of liquefaction. In the author's lecture (*Proc. Roy. Inst.*, 1896), entitled "New Researches on Liquid Air," the following observation occurs:—"The exceptionally small refractive value observed by Lord Rayleigh in the case of helium shows that the critical pressure of this body is proportionately high. It would therefore be more difficult to liquefy than a substance having about the same critical temperature but possessing a lower critical pressure than hydrogen." Now that it has been shown by Professor Moissan and the author that two substances like fluorine and argon, differing by two units in molecular weight, boil at nearly the same temperature, it seems reasonable to extend the analogy to the case of hydrogen and helium where the same difference occurs, and to suggest that they also probably have about the same volatility. If the sample of uncondensed gas resulting from the first liquefaction of the Bath gas were again treated in the same way, a much more concentrated specimen of helium could be obtained. Provided helium were wanted on a large scale, then a liquid air apparatus similar to that in use at the Royal Institution, transported to Bath and worked with the gas from the King's Well, could be made to yield a good supply. With a modified form of apparatus, it will be possible to collect any residuary gas from the use, not of 3 cubic feet of air or Bath gas, but from hundreds of cubic feet of such products. This investigation will be continued with new samples, in order to see if the composition of the gases changes and to isolate the hydrocarbons.

The author has to thank Mr. Lennox and Mr. Heath for able assistance in carrying out the experiments.

DISCUSSION.

Sir WILLIAM CROOKES said that a few days ago he received from Professor Dewar a tube containing some of the gas at atmospheric pressure. A small quantity was let into a new and completely exhausted spectrum tube, which was then re-exhausted and filled several times. On exhausting to 5 m.m. pressure and passing an induction spark it showed the nitrogen spectrum brilliantly, and on intercalating a condenser the yellow helium line was visible, but too faint to be measurable in the large spectro-scope. To remove the nitrogen, 47 c.c. were mixed in an eudiometer with an equal volume of oxygen, and sparked for about eight hours, absorption of the products being effected by strong potash solution over the mercury. When contraction had ceased, the residual oxygen was absorbed by passing pyrogallol into the potash. The unabsorbed gas amounted to 25 c.c. This gas, dried over phosphoric anhydride, was examined in a new spectrum tube, end on. (Tube shown in action). It gave the helium line (wave-length 5875.87) brilliantly, together with the other helium lines. No argon lines could be seen.

*113. "The Absorption of Hydrogen by Palladium at High Temperatures and Pressures." By Prof. DEWAR.

One of the author's earliest papers was entitled "The Motion of a Palladium Plate during the Formation of Graham's Hydrogenium." The explanation of the motion, together with a record of other experiments, can be found in the *Proc. Roy. Soc. Edin.*, 1868, vi., 504.

A subsequent investigation by the author into the physical constants of hydrogenium appeared in the *Trans. Roy. Soc. Edin.*, 1876, xxvii., 167, and had reference to the specific gravity, specific heat, and coefficient of expansion of the occluded hydrogen. These observations led to the conclusion that the specific gravity was independent of the amount of condensed gas, and had a mean value of 0.62. The specific heat, relatively to palladium of the condensed hydrogen appeared to vary inversely as the quantity occluded, but taken relatively to successive

charges was nearly constant, having the value 3.4, which is identical with that of gaseous hydrogen at constant pressure. The coefficient of cubical expansion of the alloy is about twice that of palladium, and that of the hydrogen in its compressed state not more than three times that of mercury. A later communication was made to the Philosophical Society of Cambridge (*Proc.*, 1878, iii., 207) dealing with the thermo-electric relations and electric conductivity of hydrogenium. It was shown that the potential difference of a junction of hydrogenium-palladium is at ordinary temperature nearly equal to that of an iron-copper junction, and that it increases with the temperature according to the general parabolic law; the rate of the increase being, however, greater than iron-copper, and subject to a regular variation on account of successive heatings. The formation of thermo-electric piles, and of neutral points in a wire of this substance, along with the continuous formation of thermo-electric currents through the application of a hydrogen flame were explained. Experiments on electric resistance proved that it increases directly with the amount of hydrogen condensed in the palladium.

Subsequent investigators have dealt more elaborately with the many problems suggested by hydrogenised palladium, but so far the essential facts referred to above have been confirmed.

In the course of the early observations the following experiment is recorded as illustrating the absorption of hydrogen by palladium at a red heat:—

Take a strip of thin sheet palladium, 4 or 5 c.m. long and about 5 m.m. in breadth, clamp it firmly by the end in a suitable support, so that the strip is free to vibrate, and insert it edgewise in the middle of a hydrogen flame, burning from a nozzle about 1 m.m. in diameter. If the palladium be now depressed into the inner dark cone it immediately begins to vibrate, producing a low, musical note.

If the flame be extinguished by stopping the current of hydrogen for an instant, on allowing the gas to flow the vibration commences again, and may be kept up without any actual flame.

The motion in this position in the flame is due to the absorption of hydrogen on the cool side next the inner cone, with its attendant increase of length, producing a bending of the sheet into the hot portion of the flame, where the hydrogen is instantly expelled from the palladium, which is forced to return to its original position from its natural elasticity.

It is now known that no absorption of hydrogen at atmospheric pressure by palladium takes place above 145° C., so that the cause of motion must originate at a comparatively low temperature. The question arises,—Can palladium, under any condition of pressure, absorb hydrogen at a red heat in quantity at all comparable to what it can do at lower temperatures? If free hydrogen and palladium-hydrogen are compared as regards volatility, the one boils at 30° (abs.), the other at 420° (abs.) very much like two isomeric forms of the same substance. This ratio of 1 : 14 given (and certainly the ratio could not be made greater than 1 : 16, since the absolute boiling-points may be taken as in the ratio of their respective critical points), we thus arrive at a hypothetical palladium-hydrogen critical point of 640° (abs.) or 366° C. An almost exact parallel may be drawn between palladium-hydrogen in its relation to free hydrogen, and iridium oxide in its relation to free oxygen. Thus liquid oxygen boils at 90° (abs.) and the tension of dissociation of iridium oxide is 1 atmosphere at 1423° (abs.). The ratio of the absolute boiling-points of liquid oxygen and the oxygen of iridium oxide are therefore as 1:15.9, which is almost the same value as that found above for the relative volatilities of hydrogen and palladium. In either case, the ratio of the absolute boiling-points of the respective substances may be taken as approximately representing the ratio of the latent heats of transition of state. It might then be possible that palladium no longer absorbed

hydrogen under any condition of pressure. The present experiments were undertaken with the view of answering this question.

The diagram (3) shows the general arrangement of the apparatus most suitable for examining the behaviour of the metals like palladium, sodium, potassium, &c., towards hydrogen at high temperatures and pressures.

A rod of palladium, A, weighing about 119 grms., kindly placed at my disposal by Mr. George Matthey, F.R.S., was placed in a strong steel cylinder, D, having an accurately-fitting conical joint. As little extra space as possible was left in the cylinder, which was heated in a bath of fusible metal, E. The vessel was connected with the manometer, B, by a strong copper tube, and the latter was similarly joined to a compressed gas cylinder, H, containing hydrogen. The apparatus, without the palladium, must be carefully tested at high pressures and temperatures. There must be no trace of a leak. An extra stopcock at C enabled the hydrogen accumulated in the apparatus to be blown off suddenly when required, after the hydrogen cylinder stopcock was shut off. Before commencing the experiments at high temperatures, it is well to charge the apparatus to a pressure of 20 atmospheres with hydrogen, and then blow off the gas and measure it. In this way the volume of hydrogen that is absorbed for every diminution of the pressure of hydrogen is known. In the first experiments a pressure of 20 atmospheres of hydrogen in the apparatus corresponded to 780 c.c. of gas, measured at atmospheric pressure. When the fusible metal bath was heated to 420°, and hydrogen at a pressure of 80 atmospheres introduced at starting, it fell to a pressure of 60 atmospheres in two and a half minutes. Blowing off the gas instantly to get rid of accumulated impurities, and again applying a pressure of 80 atmospheres of hydrogen, the pressure was reduced to 60 atmospheres in six minutes. When the same operations were repeated a third time, the diminution of pressure by 20 atmospheres took sixteen minutes, and a fourth operation required 28 minutes. In all, therefore, upwards of 3000 c.c. of hydrogen were absorbed in less than an hour. If the palladium could be seen at a low red heat, then during the rapid absorption of the hydrogen as described in the last experiment, the temperature must rise very considerably, and the metal, during the operation, must actually appear to grow much brighter. Calculating from the tensions of the gas, the evolution of heat at 300° must be about 4698 grm.-units of heat per grm. of hydrogen absorbed. The reverse action would take place on reducing the pressure of hydrogen in the charged palladium. After the four charges the pressure remained constant at 80 atmospheres, no more hydrogen being absorbed. The hydrogen gas outside the palladium was now suddenly blown off, the stopcock shut, and the pressure allowed to rise from the escape of gas absorbed by the palladium. In this way, it was noted that a pressure of 40 atmospheres was reached in half an hour. The whole amount of gas that had been absorbed by the metal was found, on measurement, to be 2980 c.c. After the first charge of hydrogen, the steel cylinder was opened and the palladium examined. It was found to have a deep rent in it extending along nearly the whole length of the rod. During the occlusion of the hydrogen the volume of the metal is increased by one-tenth, so that in the passage of hydrogen in and out of the metal enormous strains must be produced. As the volume of the original metal is a little less than 10 c.c., it may be taken that above 300 times its volume of hydrogen had been absorbed at the temperature of 420° and under a pressure of 80 atmospheres. The free space in the manometer and connections was now diminished, so that a pressure of 20 atmospheres corresponded to a volume of 300 c.c. of hydrogen instead of 780 c.c. as above. The palladium was saturated at 360° C. under a pressure of 80 atmospheres in the manner described above, except that a very much larger number of charges of hydrogen had to be employed. After saturation, the pressure of

hydrogen was slowly reduced to 25 atmospheres: it rose to 30 atmospheres from gas passing outwards from the metal, now heated up to 500° C., and finally reached 100 atmospheres; on cooling to 400° the pressure diminished from re-absorption of the hydrogen. On blowing off the gas between 400° C. and 500° C., 1400 c.c. of free and 3300 c.c. of combined hydrogen were found. A rod of palladium, in this way, can be quickly charged with hydrogen at about 300° C. or 400° C., and as it is only the pure gas that is occluded, this process may be used as a rapid means of getting pure hydrogen in quantity for experimental purposes.

In the next experiment, the palladium was heated to 500° C. before any hydrogen under pressure was applied. No absorption was observed till the pressure of hydrogen reached 60 atmospheres. On charging as before at pressures between 80 atmospheres and 60 atmospheres, the metal was found to absorb 1900 c.c. of gas. The experiment was repeated, with the difference that the charging pressure of hydrogen was raised to between 120 atmospheres and 100 atmospheres, and it was found that the palladium had now occluded 3700 c.c. of hydrogen. Thus it appears from these experiments that at 500° C. palladium can still occlude 300 times its volume of hydrogen under a pressure of 120 atmospheres. The observations on the tension of hydrogen in palladium by Troost and Hautefeuille showed that, for the same temperature, the values became constant and independent of the amount of occluded gas, only when the volume of hydrogen absorbed lay between 200 and 600 times that of the metal. Any other proportions gave variable tensions for the same temperature. The fact that 300 volumes can still be occluded at 500° C. seems to show that palladium and hydrogen, under such conditions, still follow the same laws of absorption as at lower temperatures. Nothing analogous to a critical point, where no combination takes place between the metal and hydrogen, has been reached.

Hoitsema published an important paper on palladium-hydrogen tensions in the *Archives Néerlandaises*, 1896, xxx., 44. In this memoir, Hoitsema gives also a series of observations on the same subject made by Roozeboom. Taking the tensions given by the latter (simply because the curve seems more regular) for the horizontal portions of the dissociation curves at different temperatures, and calculating a Willard Gibbs' formula, from the following data, viz., 20° C. pressure 7 m.m.; 100° C. pressure 205 m.m.; 170° C. pressure 1467 m.m., the expression results (where T is the absolute temperature)—

$$\log. p = 7.00338 - \frac{1983.4}{T} + 0.2378 \log. T.$$

From this it follows that the latent heat of dissociation of the palladium-hydrogen per atom of hydrogen in grm.-units is $4561 + 0.2378 T$. This would seem to show the latent heat of dissociation increases instead of diminishing with temperature. In other words, the heat of combination should be rather greater at higher temperatures, instead of diminishing as it must do if a point where no occlusion takes place were being approached. Thus theory and experiment would seem to agree.

The best and safest method for the experimental study of the relations of hydrogen and palladium at high temperatures and pressures would be to investigate the change of electrical resistance in a heated wire of the metal when subjected to different hydrogen pressures. The problem is, no doubt, more complicated, still interesting results must follow from such an investigation. Some of the electrical properties of hydrogen and palladium at low temperatures have been determined by Professor Fleming and the author, and the results will appear in future publications bearing on the subject.

The author is indebted to Mr. Robert Lennox for able assistance in the conduct of the experiments.

DISCUSSION.

Mr. R. J. FRISWELL asked whether the President had made any measurements of the tensile strength of the

steel. He was astonished to hear of the metal standing 100 atmospheres at over 500° C. He was asking for information, as he had been unable to obtain any data as to the strength of metals near a red heat, a point at which it must be rapidly falling away. The matter was of great interest for experimenters using autoclaves. Engineers did not seem to have done any work on tensile strength at points above the temperatures usual in steam boilers.

Prof. DEWAR, in reply to Mr. Friswell, agreed that no engineering formulæ existed. The experiments were dangerous, but one had to take the risk. The metal used was Whitworth compressed steel, and the vessel was made by drilling out a solid mass. He had no data as to tensile strength, the results were desired and the risk taken.

(To be continued).

PHYSICAL SOCIETY.

Ordinary Meeting, November 26th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

MR. ROLLO APPELYARD read a paper on "*The Failure of German-silver and Platinoid Wires.*"

The mechanical defectiveness, and the consequent electrical instability of alloys used for electrical wires, may be discussed from two points of view:—(1). As to the constitution and metallurgy of the alloy. (2). With regard to the subsequent treatment and environment of the wire. In stating the case, the author gives instances of the failure of German-silver and platinoid wire, that have occurred among several thousands of resistance-coils distributed over widely different latitudes. In periods of time, varying from six weeks to several years after manufacture, the wire on some of the bobbins became brittle, and broke, not only on the outer layers, but also within the coils. The towns where the faults appeared are all within the tropics, and included nearly within the isotherm of 25° C. Other coils, of nominally the same material, manufacture, and environment, have retained their original good condition. It follows that metallurgical differences exist between different samples of the same nominal quality of alloy. Examples are given to prove that failure sometimes occurs with platinoid through which no electricity has passed. Provided that the wire is good, the effect of environment is almost insignificant, *i.e.*, the question is one of metallurgy rather than of instrument-making.

The author introduces a distinction in regard to brittleness. He discriminates between "primary" and "secondary" brittleness. "Primary" brittleness is characteristic of certain alloys (for instance of gold-lead or of gold-bismuth) from the moment of their solidification. But the brittleness of German-silver and of platinoid is of a different order; it is a subsequent phenomenon. "Primary" brittleness is thus an accident of birth, and "secondary" brittleness is a disease that develops with age and circumstance. The fracture of bad specimens of German-silver and platinoid shows patches of dark metal, crevices, and fissures. It may be supposed that, during the process of cooling, "liquefaction" occurs—the metals that first solidify rejecting yet molten portions, as ice rejects foreign matter. Consequently the strength of the final alloy varies from point to point of its mass, and in passing afterwards through the die the weaker portions give way, and the general structure is loosened. Moisture can then intrude through the capillary channels. At all fissures and crevices the electric current produces undue heating: this accounts for the failure of resistance-coils on arc-light and other circuits. As regards the protection of coils against moisture, paraffin-wax is of no use whatever; it is highly absorbent. Shellac varnish is greatly to be preferred. Ebonite does not seem to have

any deteriorating effect, but it may be well to keep the alloys out of actual contact with it.

In conclusion, Mr. Appleyard expressed a hope that British metallurgists would give electrical alloys special consideration. Already British cable-manufacturers are importing thousands of tons, annually, of sheathing-wire from Germany: this is sufficiently to be regretted; he had good reason to know that instrument-makers were beginning to get the wire for their resistance-coils also from Germany. He had not enough experience of manganin to say whether that material would stand rough service in the tropics.

Prof. AYRTON said the paper had raised the extremely interesting question of the permanence of metals used for resistance-coils. Some time ago he had immersed bare platinoid wires in running water in metal tanks, and the wires all broke in short pieces. He thought, at the time, this might be due to electrolysis. On another occasion he had found that, by raising the temperature of platinoid to a dull-red heat in the air, by an electric current, any acquired faults in the wire were corrected, and the original resistance and flexibility were restored. Even when such metals are in good condition, the resistance-temperature curve does not return upon itself; it encloses a loop, indicating two distinct values for resistance at each temperature. He had been told by Dr. Muirhead that coils intended for hot climates should be enclosed in air-tight metal-cases. English manufacturers were still dubious in regard to manganin. In 1892 he had twenty coils of this material, each of 1000 ohms; the wire was silk-covered. There were 2000 volts between the terminals. Their resistance had certainly not changed by 1 in 1000, although there was some amount of vagueness regarding the fifth figure, which might be due to molecular alteration, for they were heated more than was good for resistance-coils. He confessed that this manganin had come from Germany.

Dr. S. P. THOMPSON mentioned an alloy that was proposed in Germany under the name of "Constantine." He would like to know whether any information could be obtained as to the employment of cast-iron wire. It was a metal that in some respects commended itself. He had observed the failure of some German-silver coils, but he had generally attributed it to rough handling.

Mr. W. WATSON referred to the recent work done at the Reichsanstalt with regard to German-silver and platinoid. It was there found that all alloys containing zinc were liable to erratic change of resistance, and were unsuitable for standard coils. Moreover, even the slight amount of zinc introduced into manganin during soldering with soft solder robbed that alloy of its constancy. Silver solder, containing 75 per cent of silver, should be employed for manganin. If Prof. Ayrton's coils were soldered with soft solder, that was sufficient to account for the change in the fifth figure. Shellac varnish was undoubtedly the best protection for coils. Absolute alcohol should be used as the solvent, and the coils should afterwards be heated for some hours at 140° C. If a heating-current was passed through German-silver or platinoid coils immersed in water, the general result was to produce brittleness. Mr. Watson then described a thermostat which he had contrived for drying the coils after applying the shellac varnish. A hot-air oven contains a thermometer with a platinum contact at the 140° mark and an 8 c.-p. lamp. The thermometer is in circuit with a relay actuating a mercury-key for the 8 c.p. lamp. The key consists of two mercury-cups, and a corresponding U-piece of copper, inverted, one limb to each cup. It is important to keep the heating-circuit always made; for this purpose a 32 c.-p. lamp is permanently connected between the two cups.

Prof. PERRY then read a note on a question in "*Thermodynamics*," arising from correspondence that had taken place between himself, Prof. Ramsay, and Mr. Rose-Innes, with regard to a paper in the *Roy. Soc. Trans.*

Mr. ROSE-INNES replied.

The PRESIDENT proposed a vote of thanks to the authors, and the meeting adjourned until Dec. 10th.

CORRESPONDENCE.

MOUNTING MICROSCOPIC OBJECTS.

To the Editor of the Chemical News.

SIR,—In the course of preparing the catalogue of the collection of slides belonging to the Royal Microscopical Society, I met with about a hundred anatomical preparations mounted in fluid, some in cells of considerable size. They are the work of the late R. J. Farrants, formerly a President of the Society, and were presented with his entire collection soon after his death. The date of the preparations in question is certainly previous to 1860.

I found the cells leaky, and the marine glue generally in bad condition; indeed, this was the weak point of the mounts; the cementing of the cover glasses was in nearly every case perfectly sound—as might be expected from such a master of technical details as the late Mr. Farrants.

It is certain that marine glue—so much in use many years ago for attaching cells to glass—is, in common with many other compounds of indiarubber, a somewhat unstable substance, and has evidently broken down after a period of from thirty to forty years.

The mounting fluids employed were dilute alcohol and sometimes a weak glycerin preparation, and both occasionally with some kreosote.

I have also found marine glue has failed in my own collection, and I now attach cells to glass with a mixture of red and white lead; artist's flake white, from the tube, worked up with the red lead until it is too stiff to use, and then mixed a little at a time with japanner's gold size. No more should be mixed at once than is sufficient to fix two or three cells, as the mixture rapidly becomes unworkable after the introduction of the gold size.

When the cells are in place, they should be put in a warm situation—I use the top of the hot-water cistern—and after a day or two the surplus cement should be scraped off; they are then left for a fortnight or three weeks to thoroughly harden; the final cleaning is done with a little alcohol. The cells are now impervious to ordinary mounting media.

When a cell contains an appreciable quantity of fluid, I adopt the unprofessional plan of leaving a small air bubble, which acts as a spring and saves the cell from rupture at some weak point, which sooner or later happens when cells are entirely filled with fluid.—I am, &c.,

W. T. SUFFOLK, Treas. R.M.S.

143, Beulah Hill, Norwood, S.E.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xvii.-xviii., Nos. 16-17.

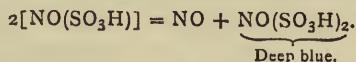
Observations on the Spectra of Compound Bodies.—A. de Gramont.—The author having noticed that many mistakes may be made by using salts, oxides, or solutions haphazard, has found that the spectra given by such compounds can be divided into two fundamental classes:—(1), the line spectra, and (2) the band spectra. The first he calls *atomic*, the second, owing to the lower temperature at which they are seen, he calls *molecular spectra*.

Amongst other interesting facts M. de Gramont finds that the proportion of an element in a compound can be recognised in three distinct manners:—1st, by a persistent spectrum, reduced to certain principal lines; 2nd, by a fleeting spectrum of unequal duration for any particular element; 3rd, by an intermittent and irregular spectrum. It should be borne in mind that the sensibility of the spectral reactions is entirely variable from one element to another.

Dissociation Spectra of Melted Salts. Alkaline Metals: Sodium, Lithium.—A. de Gramont.—Already inserted in full.

Dissociation Spectra of Melted Salts. Alkaline Metals: Potassium.—A. de Gramont.—Already inserted in full.

On Blue Nitrosodisulphonic Acid and some of its Compounds.—P. Sabaier.—In the reaction between oxide of copper and a solution of nitrate of silver the author has observed that the product, when treated with concentrated sulphuric acid, gives a very intense purple-blue colouration, which disappears on the addition of water. He finds that the principal cause of the colouration is due to the acid existing in the solution, this being due also to the presence either of nitrogen or of sulphur. In attempting the synthesis of nitrosodisulphonic acid the author tried several methods before arriving at one which now appears to be the most practicable. He effects the reaction by means of a carefully diluted sulphuric acid, previously saturated with sulphurous acid, and kept at 0°. The mixture of nitric oxide and air, when first added, gives no colouration, but after a time, varying within certain limits, there occurs a lively ebullition of the liquid, accompanied by a violent colouration, so intense as to be almost opaque. The final reaction may be expressed as—



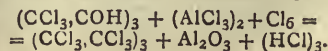
Among the properties of nitrosodisulphuric acid may be noticed that it is quickly decolourised by agitation with air, peroxide of hydrogen, or persulphuric acid; chlorine gives a similar result, as does bromine, but more slowly; iodine has apparently no effect; the alkaline chlorides are violently decomposed, with disengagement of hydrochloric acid gas and free chlorine. Sulphurous acid direct has no apparent action on the nitrosulphuric solution, even when concentrated, but when diluted by one-fifth of its volume of water the blue compound is immediately obtained. The research tends towards the preparation of the various salts of nitrosodisulphuric acid apart from the preliminary solution in concentrated sulphuric acid.

Dissociation of Minium.—H. Le Chatelier.—Experiments made with minium, free from carbonate of lime and other impurities, capable of giving off gas, show that there is still a gas evolved when heated up to temperatures varying from 445° to 636°; from which it appears that in ordinary air carrying oxygen at a tension of 150 m.m., minium can be formed at a temperature below 550°, but the temperature of most rapid oxidation is below but very close to 500°.

Impurities of Commercial Carbides of Calcium.—H. Le Chatelier.—Already inserted in full.

Action of Chlorine on Chloral in the presence of Chloride of Aluminium.—A. Mouneyrat.—The formation of the hexachlorethane is due to the fact that, by the action of chloral and chloride of aluminium, there is first of all formed a pentachlorethane, according to the equation—

$(\text{CCl}_3, \text{COH})_3 + 2(\text{AlCl}_3) = \text{Al}_2\text{O}_3 + [\text{CCl}_3, \text{CCl}_2\text{H}]_3$; this, under the influence of chlorine in the presence of chloride of aluminium, gives hydrochloric acid and hexachlorethane: the total reaction is therefore—



Action of Bromine on Chloral in the presence of Chloride of Aluminium.—A. Mouneyrat.—The author first thought that by acting on chloral in the presence of chloride of aluminium with bromine he would obtain a mixed chlorobromided compound; but he found that the body obtained did not contain any bromine in its molecule. He also tried the action of iodine under the same circumstances, but with similar results; he has obtained nothing but the hexachlorethane, C_2Cl_6 .

MISCELLANEOUS.

The Royal Society.—At the Anniversary Meeting on November 30th, the following Council and Officers were elected for the ensuing year:—

President—Lord Lister, F.R.C.S., D.C.L.

Treasurer—Sir John Evans, K.C.B., D.C.L., LL.D.

Secretaries—Prof. Michael Foster, M.A., M.D., D.C.L., LL.D.; Prof. Arthur William Rücker, M.A., D.Sc.

Foreign Secretary—Sir Edward Frankland, K.C.B., D.C.L., LL.D.

Other Members of the Council—Prof. William Grylls Adams, M.A.; Prof. Thomas Clifford Allbutt, M.D.; Sir Robert Stawell Ball, M.A.; Rev. Thomas George Bonney, D.Sc.; Prof. John Cleland, M.D.; Prof. Robert Bellamy Clifton, M.A.; Prof. James Alfred Ewing, M.A.; Alfred Bray Kempe, M.A.; John Newport Langley, D.Sc.; Joseph Larmor, D.Sc.; Prof. Nevil Story Maskelyne, M.A.; Prof. Raphael Meldola, F.C.S.; Prof. Edward Bagnall Poulton, M.A.; William James Russell, Ph.D.; Dukinfield Henry Scott, M.A.; Prof. Walter Frank Raphael Weldon, M.A.

NOTES AND QUERIES.

** Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Wheat Phosphates.—Will some correspondent kindly give me address of a manufacturer of wheat phosphates, or the name of a book or pamphlet describing the process of producing them from bran.—CATO.

MEETINGS FOR THE WEEK.

MONDAY, 6th.—Royal Institution, 5. General Monthly Meeting. Society of Chemical Industry, 8. "The Sulman-Teed Process of Gold Extraction," by H. L. Sulman and Dr. F. L. Teed.
— Society of Arts, 8. (Cantor Lectures). "Gutta Percha," by Eugene F. A. Obach, Ph.D., F.C.S.
WEDNESDAY, 8th.—Society of Arts, 8. "The Mining and Metallurgical Industries of Sweden as shown at the Stockholm Exhibition of 1897," by Bennett H. Brough.

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Professor DEWAR, M.A., LL.D., F.R.S.

Superintendent of the Laboratory:

DR. ALEXANDER SCOTT, M.A., D.Sc.

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Faraday for the purpose of promoting original research in Pure and
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belonging to the Laboratory, together with such materials and
chemicals as may be authorised.

All persons desiring to be admitted as workers, must send evidence
of scientific training, qualification, and previous experience in
original research, along with a statement of the nature of the investi-
gation they propose to undertake.

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MICHAELMAS TERM—First Monday in October to Saturday
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LENT TERM—Monday nearest to the 15th of January to the
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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1985.

ON THE ELECTROLYTIC SEPARATION OF NICKEL AND COBALT FROM IRON. ITS APPLICATION TO THE ESTIMATION OF NICKEL IN STEELS.

By O. DUCRU.

I. THE complete separation of nickel and cobalt from large quantities of iron presents considerable difficulties, and the large number of methods already published does not yet show that there is any satisfactory method known for effecting it.

The principal difficulty arises from the fact that the ferric compound (hydrate, basic acetate, &c.) always contains a considerable proportion of other metals (Ni, Co, Mn, Cu, &c.): to obtain a complete separation we are therefore obliged to multiply the precipitations, which not only takes up a great deal of time, but also leads to the accumulation of inconveniently large volumes of liquid.

II. For some years past, owing to their special properties, nickel steels have taken an important place industrially. A rapid and accurate method for the determination of nickel in these alloys is therefore of some interest. In his masterly work on the "Methodes d'Analyse Des Fers, des Fontes, et des Aciers," carried out by the desire of the Commission on the Testing of Materials for Construction, M. A. Carnot, for this purpose, gave preference to the method proposed by Rothe. This method, also suggested in France by M. Hanriot, rests, as is well known, on the separation of ferric chloride from its acid solution, by means of ether. Latterly M. Pinuera has modified this method of separation, and uses ether saturated with hydrochloric acid at a low temperature (E. Pinuera, *Comptes Rendus*, 1897, vol. cxxiv., p. 124).

III. It is easy to achieve the same result by means of electrolysis, by paying attention to the following remarks; if we precipitate a ferric solution containing, for example, nickel, with an excess of ammonia, a part of the latter metal will remain in solution, while a considerable amount is carried down with the ferric hydrate.*

If, however, we submit the ammoniacal liquid holding the precipitate in suspension to the action of electrolysis, we obtain the integral deposition of nickel on the cathode. The separation is not absolutely complete, as a small quantity of iron is also nearly always deposited on the cathode; but under suitable conditions this quantity is nearly constant; it varies from 1 to 2 m.grms., while the iron present may be about 400 or 500 m.grms. For exact work, then, it is necessary to make a correction in the weight of metal deposited: this is easily done by dissolving in hydrochloric acid, and, after peroxidation, precipitating by ammonia.

IV. The use of a nitric solution, which under analogous conditions enabled M. Riche (*Ann. Chim. Phys.*, series 5, vol. xiii., p. 528, 1878) to separate copper from iron, presents certain difficulties. It is the same with hydrochloric solutions. Good results can be obtained by using a sulphuric solution, containing sulphate of ammonia. The following is the method of procedure:—

To the solution containing the nickel and a maximum of iron is added a slight excess of sulphuric acid; it is then evaporated to dryness; the residue is taken up with

the smallest quantity of water possible, 5 to 10 grms. of sulphate of ammonia is added, and the solution warmed until it becomes clear. This solution, while agitating, is poured into the crucible of a Riche apparatus, in which 60 or 70 c.c. of concentrated ammonia has already been placed. We then proceed with the electrolysis. Two or three accumulators, coupled in series so as to obtain from 1·5 to 2·5 ampères at the commencement, form a convenient source of electricity.* Under these conditions, after about four hours, the nickel is entirely deposited.

V. This method has been checked by means of titrated solutions of iron and nickel, and some of the results obtained were as follows:—

No.	Fe m.grms.	Ni added m.grms.	Metal deposited m.grms.	Correction (iron) m.grms.	Ni recovered m.grms.	Difference m.grm.
1.	404·2	29·8	30·8	1·0	29·8	0·0
2.	269·5	74·6	75·9	1·3	74·6	0·0
3.	269·5	149·2	150·1	0·8	149·3	+0·1

The same method may be applied equally well to cobalt:—

No.	Fe m.grms.	Co added m.grms.	Metal deposited m.grms.	Correction (iron) m.grms.	Co recovered m.grms.	Difference m.grm.
4.	404·2	62·5	63·4	1·9	61·5	-1·0

VI. *Estimation of Nickel in Steels.*—250 to 300 m.grms. is dissolved in *aqua regia* in a porcelain crucible. When this is completed we add 1 c.c. of sulphuric acid, and evaporate until white fumes begin to be evolved. We then proceed as above. The following results were obtained in the analysis of a series of steels, containing from 1 per cent to 50 per cent of nickel:—

No.	Approximate quantity of present sample per cent.	Weight of sample.	Metal deposited.	Percentage found.	Correction.	Corrected percentage found.	True percentage present.
5	1	256·5	3·1	1·21	0·35	1·07	1·00
6	10	434·0	44·1	10·16	0·70	10·00	10·00
7	20	297·5	59·7	20·10	1·10	19·75	20·00
8	25	295·9	76·6	25·95	0·50	25·75	25·55
9	50	230·7	114·8	49·80	0·35	49·40	49·57

The figures given in the column *True percentage present* were obtained by a series of precipitations by ammonia in considerable excess, until the solutions no longer gave the nickel reaction with sulpho-carbonate of potash; 1 grm. of metal was used, but four or five precipitations were necessary to obtain a complete separation. The ammoniacal solutions were mixed, evaporated, and precipitated while boiling by caustic soda; the nickel was then estimated electrolytically by the method of Fresenius and Bergmann.

An examination of this table shows that in actual practice it would often be unnecessary to make the correction, a sufficient approximation being found by considering the total weight of metal deposited to be nickel; the results thus obtained, in the column headed *Percentage found* are within 0·5 per cent of being correct.† The estimation of nickel in a steel of any percentage can be effected in a few hours.

VII. Experience shows that it is unnecessary to separate the silicon or carbon; neither the small quantities of manganese and phosphorus (the sample of steel marked No. 6 contained 0·52 per cent Mn, and traces only of P) found in steels, nor the presence of chromium (the sample marked No. 7 contained 28 per cent Cr.) interfere with the use of this method; but traces of manganese are nearly always found with the small quantity of iron on the

* That is to say, 25 to 45 milliampères per square centimetre for the acting part of the cathode, assuming that the density of the current is uniform.

† Cobalt, if present, is reckoned as nickel according to the trade custom; the small quantity of copper (1 to 2 millièmes) which is generally present in nickel steels is without influence.

* This preparation reaches 27 per cent for nickel, and 48 per cent for cobalt, in the experiments described by Baumhauer (*Archives Néerlandaises*, 1870, vol. vi.), and under some conditions it may be considerably higher.

cathode. To obtain an exact correction we precipitate the two metals at once, by adding, as recommended by M. Carnot, a little peroxide of hydrogen to the solution of the deposited metals, supersaturating with ammonia, and boiling. As the coefficients of the transformation—

$$\frac{\text{Fe}}{\text{Fe}_2\text{O}_3} = 0.700 \text{ and } \frac{\text{Mn}}{\text{Mn}_3\text{O}_4} = 0.721$$

are very close, there is not room for much error in applying either of them to weights of oxides of about 2 m.grms. It is in this manner that the figures in the column marked *Correction* were obtained.

VIII. It is only natural to suppose that the small quantities of iron present on the cathode are due to the reduction, by contact with it, of the ferric and ferrous hydrates soluble in ammonia. However, no deposition is obtained by the electrolysis, under the conditions mentioned, of the ferric hydrate alone. Further, electrolysis carried on for four, or even sixteen hours, have given the same results.

On the other hand, if we add to the ferric solution notable quantities of manganese or of phosphoric acid, the proportion of iron deposited becomes considerably augmented, and the method can no longer be trusted; the traces of manganese, almost always contained in pure solutions, must therefore be carefully dealt with; as manganese easily changes from its state of oxidation, when in the near neighbourhood of the electrodes, it might without difficulty reduce some of the ferric hydrate to the ferrous state. To decide this question a ferric solution was prepared by decomposing ferrocyanide of potassium, purified by crystallisation, by sulphuric acid, and precipitating the iron in the state of sulphide: this solution was perfectly free from both manganese and phosphoric acid. It was with this that the above-mentioned experiments, 1, 2, 3, and 4, were performed, all of which gave a small quantity of iron. It is therefore probable that the presence of traces of iron on the cathode is due to a secondary electrolytic action, an action which may in time be elucidated and completely avoided.

IX. It is also necessary to remark that, although it is present in such small proportions, the iron deposited on the cathode is present in two different states; by dissolving the deposited metal in weak, warm hydrochloric acid, we can obtain a solution containing traces of iron. On the other hand, there remains a very small black residue, which is not attacked even by concentrated hydrochloric acid, but which is dissolved on boiling and the addition of a little nitric acid. The solution of this black residue in nitric acid gives the reactions of ferric salts. The extremely small quantity I was able to obtain (not more than 0.4 m.grm.) prevented me from studying it any further.

I have finally established that a very small proportion of chromic acid, in an ammoniacal solution of nickel, is sufficient to entirely stop the electrolytic deposition of the nickel, whether iron is present or not.—*Bull. Soc. Chim.*, Series 3, xvii.-xviii., Nos. 18-19.

ON THE ESTIMATION OF THE ORGANIC MATTER IN WATER BY MEANS OF PERMANGANATE OF POTASH.

By FELIX MARBOUTIN and MICHEL FRANCK.

We have estimated the amount of organic matter in solution in various waters around Paris by means of two different processes; the Albert-Lévy method as used in France, and the Forchhammer method used in England. These two methods of estimation give very different results for the same water.

M.grms. of Oxygen Absorbed to Oxidise the Organic Matter (per Litre).

	Albert-Lévy method.	Forchhammer method, modified by Sir E. Frankland.
Seine at Choisy-le-Roi ..	2.92	1.53
" Ivry	3.00	1.66
" Austerlitz	2.60	1.27
" Argenteuil	2.43	1.26
Marne at Neuilly	1.22	0.69
" Saint-Maur	1.13	0.56
Ourcq at the Vilette basin ..	1.46	0.82
Ayre reservoir, Rue Villejust ..	0.24	0.08
Drain at Epinay (Gennevilliers)	1.13	0.39
Drain at Gresillons (Gennevilliers)	1.05	0.36
Drain at Garene (Achères) ..	0.65	0.24

This table shows that, for river waters, the French method gives figures double those of the English method; while for certain springs and surface drainage waters the numbers are tripled.

In view of this fact, it may be interesting to compare the tables as arranged in England and France for the classification of waters by the estimation of organic matter.

M.grms. of Oxygen Absorbed to Oxidise the Organic Matter (per Litre).

Nature of the water.	FRANCE. Albert-Lévy process.	ENGLAND. Sir E. Frankland by the Forchhammer process.	
		Surface waters from uncultivated land.	Surface waters other than from uncultivated land.
	M.grms.	M.grms.	M.grms.
Very pure ..	Less than 1.0	Less than 1.0	Less than 0.5
Medium purity ..	1.0 to 2.0	1.0 to 3.0	0.5 to 1.5
Suspicious ..	3.0 to 4.0	3.0 to 4.0	1.5 to 2.0
Impure	More than 4.0	More than 4.0	More than 2.0

Thus it appears that river waters are classified in the same category both in France and England, in spite of the difference in the results; but such is not always the case with springs and surface drainage water. In France, the Commission on Hygiene did not think it worth while to make any difference between surface waters from uncultivated or cultivated sources, and it is noticeable that they are more strict than in England for springs and surface drainage waters.—*Bull. Soc. Chim.*, Series 3, vols. xvii.-xiii., Nos. 18-19.

NOTE ON A SOMEWHAT REMARKABLE CASE OF THE RAPID POLYMERISATION OF CHLORAL.

By J. W. MALLET.

A SPECIMEN of anhydrous chloral—trichloroacetaldehyde—of about 250 grms, was contained in a glass vial of a form commonly used in Germany, with small drawn-out neck, hermetically sealed. It had been on hand for more than a year, and had originally been quite clear, but doubtless not absolutely pure, very likely containing traces of sulphuric acid, as a few small white flocks of the polymeric meta-chloral had begun to show at the bottom.

This specimen, with a number of others, had been exhibited on the lecture-table, handled by several persons, and moved back to the collection-room whence it had come. It was placed temporarily on a table, among other bottles, in the afternoon, to be next morning

restored to its place in the collection on the shelves. No one went into this room, which is in a locked portion of the building, before the following day. The temperature was moderate—about 20° or 22° C.—and differed but little from that of the lecture-room. The place on the table occupied by the bottles did not receive any direct rays of the sun.

On coming into the room in the morning a strong odour of chloral at once drew attention to the table, where a circle of half a metre or so in diameter was covered with a thin crust of white meta-chloral. The remains of the sealed vial were found as small fragments scattered about the room, some of them as much as two or two and a half metres away from where the vial had stood, and some of them on the shelves at a higher level than the top of the table, showing clearly that the vial had not fallen down, or been knocked over by a mouse or rat, but had burst, and with some considerable force, although no other bottle standing near it had been broken.

It seems that polymerisation must have taken place so rapidly, and to so large an extent, that the heat evolved in the union of the smaller into larger molecules raised the temperature of the remaining liquid chloral to a point at which the tension of its vapour was more than the vial could withstand.

If this explanation be accepted, the occurrence is in three respects noteworthy. In the first place, the amount of heat evolved in the polymeric change must have been quite large to produce the effect observed, remembering that liquid chloral is only about as volatile as water—has about the same boiling-point under atmospheric pressure; I do not know of any recorded measurements of the thermal value of the change in question. Secondly, the polymeric change must have occurred with a degree of suddenness which is surprising in view of the very gradual transition from liquid chloral to meta-chloral which is usually observed; other specimens, prepared in this laboratory, have taken several years to become entirely solid. Thirdly, it is not easy to imagine what cause can have provoked this sudden polymerisation, there having been less mechanical agitation, less change of temperature, less external disturbance of any kind that can be pointed out, at the time when the change occurred than the specimen had previously been exposed to.—*American Chemical Journal*, xix., No. 9, Nov., 1897.

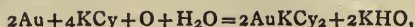
NOTE ON THE

SO-CALLED "SELECTIVE ACTION" OF CYANIDE OF POTASSIUM FOR GOLD.*

By W. A. DIXON.

FOR some years past there has been considerable discussion as to a "selective action" exhibited by very much diluted solutions of cyanide of potassium for gold, so that it passes into the liquid as aurocyanide of potassium, whilst the base metals are left behind in the ore mass during treatment by the cyanide process. This is held to be the reverse of what occurs when a more concentrated solution of cyanide is employed, as in that case a large proportion of base metal in comparison to the gold is obtained in solution. I think the following considerations will show how this occurs, and that there is really no such thing as "selective action." By a selective action it seems to be generally understood that in the weak solution the action is the reverse of what it is in a strong one, an idea of volition on the part of the cyanide being implied in the statement.

The reaction by which gold is dissolved by cyanide of potassium is—



the oxygen being supplied by that present in solution in the water, or by oxidising agents, as chlorine, bromine, ferricyanides, peroxides, &c.; in practice the first is the usual source.

A ton of ore in powder requires from 100 to 110 gallons of water to thoroughly wet it, or, say, an average of 105 gallons, which at 70,000 grains per gallon is equal to 7,350,000 grains. The coefficient of solubility of oxygen at 30 ins. barometric pressure and 60° F. is 0.00295, which, divided by 5 to reduce it to that due to the partial pressure of oxygen in air, gives as the coefficient of absorption of oxygen from air 0.00059, so that 105 gallons of water would contain 43,383 grain measures of oxygen in solution, which would weigh 62 grains. These 62 grains of oxygen contained in 105 gallons, as a maximum, would, in conjunction with cyanide of potassium, dissolve 1527 grains of gold, that is 3 ozs. 3 dwts. 15 grs., and the quantity of cyanide required would be 1001 grains, so that the solution would be very dilute, containing only 0.0047 per cent of cyanogen.

It appears clear to me that we have here a sufficient explanation of the so-called "selective action." Gold in presence of oxygen has in all cases a superior affinity for soluble cyanides, or the soluble cyanides have for gold, than the soluble cyanides have for the compounds of the base metals. These compounds have the same affinity for cyanogen whether oxygen in the free state or easily available is present or not, whilst if oxygen is absent the action on gold is *nil*. The base metals always occur in gold ores as oxides, sulphides, or other compounds, whilst the gold is in the free state. In these circumstances the gold is first dissolved as long as free oxygen is available, and then, oxygen being exhausted, base metals pass into solution until either they are exhausted or the cyanide is saturated, whichever happens first, a sufficient time being given to complete the reaction. A much diluted solution of cyanide contains much free oxygen in proportion to the cyanide, and therefore dissolves much gold in proportion to the cyanide present, and afterwards little base metal, because there is little cyanide left to saturate, and it therefore appears that the cyanide has selected the gold. In a solution containing more cyanide, gold is dissolved till the oxygen is exhausted, and then the excess of cyanide enters into double decomposition with the compounds of the base metals, which are then found in solution in greater proportion relatively to the gold, and the solution appears to have had a selective action on them.

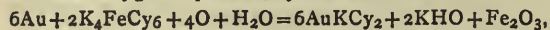
Some metals in the free state, as zinc, for example, have a superior affinity for cyanogen to gold, but these do not occur free in ores, but when a solution containing gold as cyanide is brought in contact with one of them, as zinc, it dissolves, and the gold is precipitated. Zinc, which is commonly used for this purpose, is moreover known to precipitate gold the more efficiently the more free it is from compounds such as its oxide.

In practical work the oxygen in solution in the water used would be much less than that indicated, as it would be removed by organic and mineral substances undergoing oxidation, but, on the other hand, minerals treated by the cyanide process have usually much less gold than 3 ozs. per ton, or they are treated several times with fresh solutions. This re-treatment is simply a renewal of the conditions favourable to the solution of gold, an oxygen-holding solution of cyanide replacing one which is exhausted of that element.

In the current literature on this subject two distinct sets of reactions have become mixed up, and are spoken of as one, the substances which caused them being called "cyanicides." This word should be confined to those substances which by their action on cyanides decompose them, or set free hydrocyanic acid, which soon decomposes. These substances do not combine with cyanogen

* Read before the Institution of Mining and Metallurgy, Nov. 17th, 1897.

or alkaline cyanides; they kill them, and to such the word cyanicidic is truly applicable. On the other hand, "cyanicidic" is not applicable to substances which by entering into combination with cyanogen or cyanides may be rather regarded as causing sleep rather than death, and it is only on these that the so-called "selective action" can be exercised, and the compounds of only three of these are found in gold ores, namely, iron, copper, and zinc. Iron present in the ferric state may be inert or act as a cyanicidic if present as a salt, *i.e.*, as ferric sulphate; in the ferrous state it combines with cyanide of potassium to form ferro-cyanide of potassium, but this also dissolves gold if sufficient oxygen is present, by the reaction—



and therefore it is not entitled to a position amongst the metals which may be selected. These are therefore reduced to zinc and copper, and the compounds of these are only dissolved in absence of sufficient oxygen with the cyanide of potassium to dissolve the gold. In most gold ores these metals are absent, and then the question of selection does not arise, and solutions of cyanide of any strength may be used, but diluted ones would be most convenient, as they supply the necessary oxygen and consume less cyanide. It is to be supposed that sulphates or other cyanicidic compounds are removed or decomposed by a preliminary treatment.

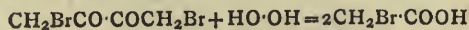
In practice it must necessarily happen that solutions containing more than the theoretical quantity of cyanide to the quantity of oxygen present must be used, even when cyanicidic are absent, in consequence of the gold being distributed in granules throughout an immense mass of ore. A granule of gold exhausts the cyanide and oxygen in its immediate neighbourhood, and action then ceases until a fresh supply of both arrives by diffusion from the surrounding solution. I do not know that the relative rate of diffusion of oxygen and cyanide in watery solution has been determined, but it must be that 16 of oxygen will diffuse sooner than 260 parts of cyanide of potassium, and therefore an excess of cyanide must be used to compensate for this difference.

SOME NEW DERIVATIVES OF DIACETYL.

By HARRY F. KELLER and PHILIP MAAS.

SINCE communicating the results obtained in studying the action of oxidising agents upon diacetyl, we have continued our work on this diketone in several directions.

The ready and quantitative conversion of diacetyl into acetic acid by certain oxidising agents, notably hydrogen peroxide, induced us to try whether the halogen-substituted products, dibromo-diacetyl and tetrabromo-diacetyl, would behave in an analogous manner. If they, respectively, yielded bromacetic acid and dibromacetic acid, their symmetrical structure might be regarded as definitely established. Thus:—

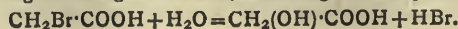


and—



While both compounds were found to be readily attacked by the oxidising agent, only the dibromo-derivative gave something like the expected result.

A weighed quantity of this substance was treated in the cold with a moderate excess of hydrogen peroxide solution. The greasy scales gradually disappeared, and the liquid became strongly acid. Upon evaporating, it gave off hydrobromic acid, and left a residue consisting of a mixture of bromacetic and glycollic (oxyacetic) acids. This mixture was completely converted into the latter acid by prolonged boiling with water, according to the equation—



The characteristic calcium salt was prepared. It was obtained in stellar aggregations of needles, having a silky lustre, insoluble in alcohol and only sparingly soluble in cold water. It contained:—

	I.	II.	Calculated for (C ₅ H ₇ O ₂) ₂ Ca + 4H ₂ O.
Ca	15.06	15.54	15.26
H ₂ O	26.97	26.47	27.48

The salt is understood to be somewhat efflorescent, and the percentage of water therefore a little too low. The dehydrated salt gave Ca = 21.32 per cent, instead of 21.05 per cent required by theory.

The blue copper salt was also obtained.

A mixture of several acids is produced when dibromo-diacetyl is heated with nitric acid. We have not been able to effect their separation, but have reasons to believe that oxalic acid and monobromacetic acid are amongst them.

When tetrabromodiacetyl was treated with hydrogen peroxide, its yellow colour disappeared slowly. A white flocculent precipitate formed while the dense powder dissolved. The colourless solution had a peculiar penetrating odour, and deposited, upon standing, another crop of the product in the form of long colourless needles with a silky lustre. From 3 to 4 grms. of this substance were thus obtained from 10 grms. of the diketone.

The mother liquor, still having the characteristic odour of the crystallised product, was extracted with ether, but the latter, on evaporating, left only a small quantity of an oily liquid.

The crystals were found to be insoluble in water, and very freely soluble in alcohol, ether, benzene, glacial acetic acid, and other organic solvents. From an ethereal solution very beautiful prismatic crystals, more than a centimetre long, were obtained. They belong, without doubt, to the rhombic system, and consist of several prisms, the orthopinacoid and a brachydome. From the solution in acetic acid the substance is re-precipitated in the form of long fine needles when much water is added. It melts at 72.8°, and at higher temperatures sublimes, apparently without decomposition.

Analyses of the carefully purified substance yielded:—

	I. Per cent.	II. Per cent.	Calculated for C ₅ HBr ₂ O. Per cent.
C	8.13	8.04	7.94
H	0.56	0.26	0.22
Br	87.99	88.04	88.30
O	[3.32]	[3.66]	3.53

Determinations of the molecular weight by the cryoscopic method, by means of Beckmann's apparatus and glacial acetic acid as the solvent, gave—

	I.	II.
Solvent	19.485 grms.	19.663 grms.
Substance	0.1788 gm.	0.3360 gm.
Depression	0.084°	0.146°

I.	II.	Calculated.
461	435	452

The composition and molecular weight correspond to the formula C₅HBr₂O, which is that of *pentabromacetone*. That this is really the compound we obtained is further proved by a comparison of the physical properties* with those observed by other chemists, as well as its behaviour

* The melting-point is given by Beilstein (3rd ed., p. 989) as 76°. Although we repeated the determination several times, using material purified with scrupulous care, and the best thermometers, it invariably gave the same result, 72.8°.

upon boiling with barium hydroxide; bromoform was produced and was identified, after distilling it in a current of steam, by its odour and boiling-point, and the formation of phenylcarbylamine when the alcoholic solution was treated with aniline and caustic potash.

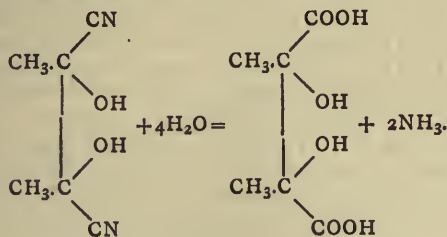
It is remarkable that an acetone derivative should result from the oxidation of a diketone, for methyl-ethyl ketone can be converted by oxidation into diacetyl (Fileti and Ponzio, *Gazz. Chim. Ital.*, xxv., 233).

The oily product mentioned above, and which was extracted from the solution after the pentabromacetone had separated, may have been tribromacetone. It had a powerful odour, decomposed upon heating, and did not respond to the carbylamine reaction.

Attempts to oxidise tetrabromodiacetyl with nitric acid have not as yet given any definite results. Although the substance dissolves rapidly on warming in the concentrated, especially the red fuming, acid, the greater part of it separates again on cooling or diluting with water in large yellow crystals.

Cyanohydrins.—Another class of diacetyl derivatives to which we have lately given some attention are the cyanohydrins.

Fittig and one of us have shown that diacetyl and hydrocyanic acids unite quantitatively to form a dicyanohydrin, and that this may be readily converted into dimethylracemic acid.



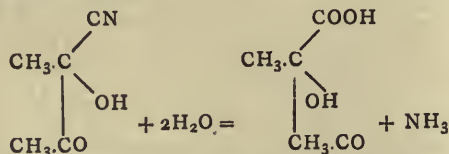
Our recent experiments were undertaken—

1. To attempt the preparation of a monocyanohydrin of diacetyl.

2. To produce halogen-substituted cyanohydrins.

If we succeeded in converting the latter into the corresponding acids, we would have the first derivatives of tartaric acid in which hydrogen is replaced by halogen.

Diacetyl and hydrocyanic acid (30 per cent solution) were brought together in molecular proportions. A rise of temperature and the vanishing of the yellow colour indicated that combination was taking place. The liquid was gently heated, and then allowed to stand for some time in a warm place. After extracting with ether, and evaporating this solvent, there remained a colourless viscous liquid. This was placed over dehydrating materials, and repeatedly cooled to very low temperatures without showing any signs of crystallising or congealing. It was doubtless the expected compound, though probably very impure. We now attempted to produce the corresponding acid—



by treatment with strong hydrochloric acid. This was tried in a number of ways; but although even the most concentrated acid (saturated at 0°) had no appreciable effect at ordinary temperatures, the slightest warming destroyed the compound, with formation of dark resinous products, from which an acid of definite composition could not be isolated.

More encouraging were the results of our experiments

upon dibromodiacetyl. It was found to combine with hydrocyanic acid quite readily, and yielded a well-crystallised dicyanohydrin. From the ethereal extract it was obtained in fine colourless crystals, having a strong lustre, and melting with decomposition at 177°.

Its composition is established by these determinations:—

	Found.		Calculated.
	I. Per cent.	II. Per cent.	Per cent.
N	9.38		9.39
Br.. ..	54.35	54.74	53.69

The excess of bromine is probably due to the formation of calcium cyanide when the substance is ignited with lime.

Experiments are in progress to transform the nitrile into the acid; the amide of the acid has been obtained in small quantities.

We have also found that tetrabromodiacetyl can be made to combine without difficulty with two molecules of hydrocyanic acid. The resulting body seems to be analogous to the tetrachlorodiacetyl dicyanohydrin described by Levy and Witte (*Ann.*, ccliv, 98). As yet, however, we have not prepared a sufficient quantity to warrant the publication of definite data.

Dichlorodiacetyl.—As Levy and his pupils (*Ann.*, ccxlix., 93) obtained their tetrachlorodiacetyl by a complicated process, the action of potassium chlorate and hydrochloric acid on chloranilic acid, we have tried to prepare it by direct chlorination of diacetyl. It was found that at first the action of chlorine upon the diketone, dissolved in carbon disulphide or chloroform, is quite energetic, but the only product we have so far obtained proves to be dichlorodiacetyl. It closely resembles the dibromo-derivative; soluble in carbon disulphide, chloroform, boiling petroleum ether, and warm benzene, it crystallises in yellowish unctuous scales melting at 124.5°. A chlorine determination gave—

	Found.	Required for
	Per cent.	C ₄ H ₆ -I ₂ O. Per cent.
Cl	45.63	45.80

—*Journal of the Franklin Institute*, vol. cxliv., No. 5,

A CONTRIBUTION TO THE STUDY OF OXYGEN AT LOW PRESSURES.*

By R. THRELFALL, M.A., Professor of Physics in the University of Sydney, and FLORENCE MARTIN.

WHEN a mass of oxygen is enclosed in a tube and the mercury pressure on it continuously diminished, it is found that at about 0.7 m.m., and over a certain range of lower pressures, the gas appears to undergo a change of condition. The phenomenon may be described in the words of Bohr, its discoverer (*Wied. Ann.*, xxvii., p. 475). "A given mass of oxygen is enclosed in a tube, and the mercury adjusted so as to give to a pressure rather less than 0.7 m.m. If the volume of the gas is now reduced by raising the pressure, say to 0.8 m.m., it is noted that this pressure will not remain constant, but varies more or less with lapse of time. In three to five hours the pressure will fall by some 12 per cent of its initial value (the volume being constant). After five hours the pressure was found to have attained its steady value, so far as observations extending over twenty-four to thirty-six hours could determine."

This curious behaviour of oxygen was also noted by Baly and Ramsay (*Phil. Mag.*, xxxviii., p. 324, 1894), who observed that at a pressure of about 0.75 m.m. oxygen becomes unstable as to its pressure-volume relation, and

* Read before the Royal Society of N. S. Wales, June 2, 1897.

that the equilibrium condition is not attained until after seventy-eight hours rest. The slightest change of pressure or volume then upsets the equilibrium, and time has again to elapse before a steady state is attained. It appears likely either that the oxygen forms an allotropic modification or that it forms some compound with mercury or other material present and with which it is in contact.

It will be noted that, according to Bohr, the volume of the gas tends to increase below 0.7 m.m., indicating that the molecules of oxygen are partly split up. In this case, therefore, it would be reasonable to infer an increase of oxidising power, and it is possibly to this cause that the soiling of the fall-tubes of Sprengel pumps is to be attributed. It appears worth while, therefore, to try to arrange some chemical test capable of showing the presence of active oxygen.

The two following test solutions were found to satisfy the conditions, though one was more sensitive than the other. One condition of course is that the test solution must not have a vapour-pressure comparable with 0.7 m.m. The first indicator tried was a solution of indigo in pure sulphuric acid. This is bleached by ozone, but experiment showed that the reaction does not afford a very delicate test of the presence of that gas. Another solution was therefore tried, consisting of potassium iodide and starch dissolved in glycerin. The glycerin was carefully dried at a temperature of 260° C. When cool, some of it was mixed with a small quantity of powdered potassium iodide. A very small quantity of starch was added to the remainder of the glycerin, which was then slowly heated till the starch was quite dissolved and the liquid again became transparent. When cold, this portion was mixed with the potassium iodide solution—a solution so prepared is not affected by ordinary oxygen, but one bubble of the gas which has passed through an ozone tube turns it bright yellow, and three bubbles give it a dark blue, almost black colour. This seemed sufficiently sensitive, and was accordingly adopted. Of course the starch is not absolutely necessary, iodine being liberated in large enough quantities to colour the solution, but it was considered to be of some advantage to use it as an additional verification. Oxygen was prepared and purified in the usual manner, and stored in a gas-holder.

On leaving the gas-holder the gas passed through (1) a system of purifying tubes containing (a) nitrate of silver, (b) solid potash, (c) sulphuric acid, (d) phosphorus pentoxide; (2) a wash-bottle containing a small quantity of the potassium iodide solution; (3) an ozoniser by which the oxygen could, when required, be ozonised without altering any of the apparatus.

Two diagonal glass taps, in series, allowed the purified gas to pass into the exhausted part of the apparatus. This consisted of a glass tube about 0.2 c.m. in diameter and 30 c.m. long, to which was fixed a mercury pressure gauge of the U type, 0.1 c.m. in diameter. In order to prevent a possible loss of active oxygen through the action of the mercury in the gauge, the latter was connected to the exhausted space by a capillary connection.

The exhausted tube was connected through a small wash-bottle with a Fleuss pump, the wash-bottle containing a small quantity of the sensitive solution. A similar wash-bottle, containing the same solution, was arranged to stand close to the bottle through which the gas was passed in order to enable colour comparisons to be made. All the apparatus was, practically speaking, either fused together or had joints protected by paraffin and mercury, the use of indiarubber being of course inadmissible. The Fleuss pump was worked by an electric motor, and the taps were adjusted until a steady stream of oxygen could be passed through the apparatus at a pressure of about 0.25 m.m. of mercury.

The apparatus, after having been made entirely airtight, was filled with oxygen and exhausted several times; a steady stream of oxygen, at atmospheric pressure, was then run through it for an hour, in order to get rid of

traces of air. It was then exhausted and kept at a constant pressure of nearly 0.25 m.m. (never less than 0.1 nor greater than 0.4 m.m.), with the oxygen bubbles coming through at the rate of twenty per minute. Each bubble of oxygen, on reaching the exhausted tube, was therefore reduced in pressure over the range of instability. After six hours, no change having taken place in the potassium iodide solution, the apparatus was filled with oxygen at atmospheric pressure and left for several hours. This experiment was repeated during three days; that is to say, the oxygen was passing through the apparatus at a pressure of 0.25 m.m. for 17.5 hours altogether. At the end of this time, no trace of the ozone reaction being observable, it was considered advisable to ascertain whether if a very small proportion of the oxygen passing through had become converted into ozone, so minute a quantity, at so low a pressure, would affect the test solution. With this object, the wires of the ozoniser were now joined up, and it was found that in one minute a faint yellow colouring of the solution, slight but distinctly visible, occurred. Evidently, therefore, twenty bubbles of electrically ozonised oxygen produce more effect than 21,000 bubbles of oxygen which has been simply subjected to the effects of low pressure. And even if the experiment described above is not considered to prove, with sufficient conclusiveness, that low pressure alone has no power to cause the formation of ozone in oxygen, it must at least be admitted that the ozone so formed is less than 1/1000th of the quantity produced by an ozoniser in the ordinary way in the same volume of oxygen, and as this can scarcely exceed 5 per cent of the whole volume, the ozone formed by lowering the pressure cannot be so much as 0.005 per cent of the volume of oxygen present.

We must not neglect to state that our curiosity in this matter was stimulated to the experimenting point by a letter from our friend, Mr. W. Sutherland, of Melbourne, who considered, on grounds based on the kinetic theory of gases, that allotropic oxygen of some kind would most likely be found at about the pressure we employed. The soiling or Sprengel pumps, however, as well as the experiments of Baly and Ramsay, had previously led us, independently, to infer the possibility of a production of active oxygen under the conditions we have mentioned.

A REVISION OF THE ATOMIC WEIGHT OF NICKEL.*

FIRST PAPER.—THE ANALYSIS OF NICKELOUS BROMIDE.

By THEODORE WILLIAM RICHARDS
and
ALLERTON SEWARD CUSHMAN.

Introduction.

OF the atomic weights to-day those of nickel and cobalt are among the most interesting, not only because the two values are so close together, but also because the purely elementary character of these metals has been recently doubted (*Zeit. Anorg. Chem.*, ii., 235; *Berichte*, 1889, xi., 2026). A careful study of the available literature upon the subject leaves the impartial critic in grave doubt as to the true values to be accepted, hence an experimental revision seems to be imperative; and the present work is a part of a comprehensive attempt to conduct such a revision. The very useful and complete index of the work which has been done upon this subject, recently published by Professor Clarke (*"Recalculation of the Atomic Weights," Smithsonian. Misc. Coll., Constants of Nature, Part V., 1897, p. 291*), makes a detailed statement of the various researches unnecessary here; but a chronological list is appended.

* Contribution from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, vol. xxxiii., No. 7.

The Atomic Weight of Nickel.

O = 16.

1826.	By Analysis of Chloride.	
	Rothoff	59.1
1852.	Reduction of the Oxide by Hydrogen.	
	Erdmann and Marchand	58.2—58.6
1856.	Conversion of Nickel to Sulphate.	
	Deville	58.85
1857.	Analysis of the Oxalate.	
	Schneider	58.07
1858.	Analysis of the Sulphate and Chloride.	
	Marignac	58.4—59.29
1860.	Synthesis of the Chloride.	
	Dumas	59.02
1863.	Reduction of the Oxides in Hydrogen.	
	Russell	58.74
1866.	Analysis of Nickel Potassic Sulphate.	
	Sommaruga	58.03
1867.	By relation to Gold, out of Sodic Auro-Chloride.	
	Winkler	59.45
1867.	Measurement of Hydrogen evolved by acting on Nickel with HCl.	
	Russell	58.77
1871.	Analysis of Strychnine and Brucine Nickelocyanides.	
	Lee	58.01
1883.	Analysis of the Sulphate.	
	Bauby	58.73
1886.	Reduction of Oxide (NiO) by Hydrogen.	
	Zimmermann	58.71
1890.	Mond, Langer, and Quincke	58.58
1892.	Conversion of Sulphate to Oxide, and Reduction in Hydrogen.	
	Schützenberger	58.54
1892.	Krüss and Schmidt	57—64
1893-94.	Analysis of Nickelous Chloride and by relation to Iodine.	
	Winkler	59.05—58.87
	Value accepted by Ostwald, 1890.. (Lehrbuch, i., p. 96).	Ni = 58.5
	Value accepted by Clarke, 1896 .. ("Recalculation of the Atomic Weights," loc. cit.).	Ni = 58.687
	Value accepted by Seubert, 1896 .. (Zeit. Anorg. Chem., xiii., 229).	Ni = 58.9

by securing unanimous results from a number of compounds and methods, it is equally obvious that a single method, well worked out, is far better than twenty incomplete ones; hence the present work was confined for the present to a single compound.

In our choice of material, we were guided by experience already gained in this Laboratory. The advantage of the bromides as typical compounds in atomic weight determinations has been discussed by one of us in preceding publications, and requires no further mention. The rather meagre current descriptions of the properties of anhydrous nickel bromide did not seem encouraging, but the progress of the research showed that it is possible to dry this salt, weigh it, and dissolve it in water in a perfectly normal condition. Since this was the case, nickelous bromide was naturally chosen as our starting point.

The Preparation and Properties of Nickelous Bromide.

Finely divided nickel, when heated to a red heat in a stream of dry bromine vapour, readily forms the bromide, which sublimes at bright redness. The colour of the sublimate varies from a pale straw-yellow to a dark bronze-brown, according to the state of aggregation. At a red heat in the presence of traces of air or moisture, the salt loses traces of bromine with the formation of bright green nickelous oxide, unless much hydrobromic acid is present. We have never obtained evidence that an oxybromide is formed under these conditions.

The sublimed bromide is almost insoluble in cold water, but solution soon becomes apparent to the eye in water at 50°. In water at 90° the salt dissolves less slowly, but a grm. still requires an hour or two for its complete solution. When originally free from oxide, the sublimed bromide dissolves in water even at the boiling point to form a solution of perfect clearness. According to Berthelot (*Anal. Chim. Phys.* [2], xlv., 389) a solution of nickelous bromide left for some time in contact with air deposits some flakes of the oxide. We have never met with the slightest evidence of the truth of this statement in the case of a dilute solution. If, however, the nickelous bromide contained only a slight admixture of the oxide, this oxide might escape observation until it had settled out upon the bottom of the vessel. Dilute nitric acid does not materially hasten the solution of the bromide. The sublimed crystalline salt is hygroscopic in character, although not nearly to the extent which we had been led to expect. From several experiments carried out to test

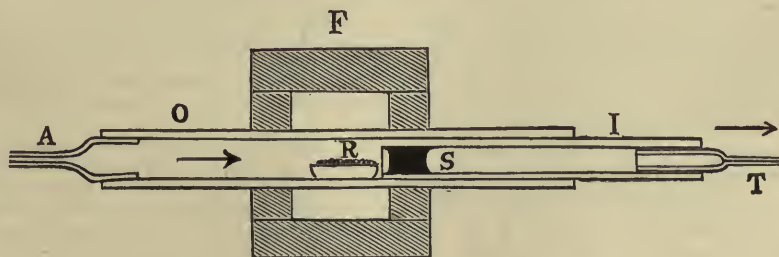


FIG. 1.—SECTION OF APPARATUS FOR SUBLIMATION.

A, Glass tube for admitting bromine vapour. O, Outer porcelain tube. I, Inner porcelain tube. T, Glass outlet tube. F, Perforated Fletcher furnace. R, Boat containing nickel. S, Sublimed bromide.

A glance at this list shows the lack of consistency in the results obtained. In many cases this is sufficiently accounted for by the inadequacy of the methods and the known impurity of the materials used, without taking into consideration the unconfirmed admixture of unknown impurities. A critical investigation of the data already published would be an endless and perhaps unprofitable labour, and we prefer at this time rather to record the method and results of our own work than to enter into any discussion of claims heretofore advanced.

While it is obvious that certainty is to be obtained only

at this point it appeared that freshly sublimed nickelous bromide exposed to the free air of the room absorbed about 0.1 milligram. per grm. in ten minutes.

Since the value of the selection of nickelous bromide as the starting point in the determination of the atomic weight of nickel depends on the possibility of dissolving the salt in hot water without the slightest loss of bromine, it seemed necessary to investigate this point. To this end, a mass of several grms. of the sublimed compound was suspended in a flask arranged in such a way that the atmosphere of the flask during the solution could be

swept through a bulb-tube containing a mixture of potassium iodide and starch. Any formation of nickelous oxide or oxybromide during the heating of the solution could have taken place only by disengagement of bromine, which would have been shown by the blueing of the indicator in the bulb. Not the slightest tinge of blue colour appeared at any time during the experiment, in spite of the fact that the solution of nickel bromide was finally subjected to prolonged boiling. That the iodo-starch mixture was exceedingly sensitive to very minute traces of free bromine was assured by experiment in the first place. This experiment is detailed here, however, merely as corroborative testimony, and is not advanced as being final on the subject; the best evidence is yielded by the quantitative results of our series of analyses.

Being now sure that we could analyse the salt if we could obtain it in a pure state, we next proceeded to determine whether it could be prepared in a condition altogether suitable for weighing and analysis. The first sublimations were performed in an ordinary combustion tube in an atmosphere of carbon dioxide. These sublimations yielded in no case material that was above suspicion, and none of it was used in our analyses, the experiments being carried out merely as a study of the properties of the substance under examination. Since it seemed possible that the carbon dioxide used might suffer partial dissociation at the high temperature necessary for the sublimation, nitrogen was substituted for this gas. After many experiments with nitrogen, both alone and when mixed with bromine vapour, it was found best to carry on the sublimation of the nickelous bromide in a stream of nitrogen mixed with hydrobromic acid. The elaborate apparatus constructed by Mr. G. P. Baxter for supplying suitable mixtures of any or all of these gases and vapours for preparing and subliming the salt will be described in a later paper, upon the atomic weight of cobalt.

The temperature at which the salt sublimes lies not far from that at which the hardest glass begins to soften, hence we found it advantageous to conduct the sublimation in porcelain. In order to collect the product a small porcelain tube, used as the receiver, was fitted, telescope fashion, inside of the larger tube containing the substance to be vaporised. In this way the pure crystals could be removed without contamination. Since nickelous bromide is decomposed either by oxygen or by water at a red heat, unless a very large excess of hydrobromic acid gas is present, both oxygen and water must be excluded with scrupulous care during its final sublimation. At first we were not able to accomplish this complete exclusion, so that most of the bromide used in the preliminary series of analyses contained traces of green crystalline oxide, which were carefully collected and weighed. In making the final calculation, the weight of this oxide was subtracted from the total, in order to obtain the weight of the bromide. Subsequently, with improved apparatus, the bromide was obtained in a state of satisfactory purity; it gave a perfectly clear solution in water, leaving nothing to be desired. With the improved arrangement the oxide itself was quickly converted at a dull red heat into the bromide, by means of a stream of hydrobromic acid gas. Hence it was possible to purify nickelous bromide contaminated with oxide by simply treating it for a few minutes in this fashion; in practice, the scheme worked very well.

Chemical literature contains no satisfactory determination of the specific gravity of the anhydrous salt; hence, in order to reduce our weighings to the vacuum standard, the following determination was made:—3.3196 grms. of pure dry nickelous bromide displaced upon one occasion 0.6162 grm. and upon another 0.6156 grm. of rectified toluol at 28°. Since the specific gravity of the toluol at this temperature was 0.860, referred to water at the same temperature, the specific gravity of the bromide must be 4.64. Nickelous bromide is insoluble in toluol.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, November 4th, 1897.

(Concluded from p. 276).

114. "On some Yellow Vegetable Colouring Matters." By A. G. PERKIN.

The *Rhus rhodanthema*, a tree growing to the height of 70 or 80 feet, is indigenous to northern New South Wales. The colouring matter $C_{15}H_{10}O_6$ is identical with fisetin. A glucoside of fisetin, $C_{36}H_{30}O_{16}$ ($C=60.18$; $H=4.45$), colourless needles, m. p. $215-217^\circ$, is also present; it is decomposed with difficulty by boiling dilute acids. This closely resembles fustin, $C_{38}H_{46}O_{23}$ or $C_{36}H_{26}O_{14}$ ($C=63.34$; $H=3.81$), m. p. $217-219^\circ$, the fisetin glucoside of *R. Cotinus* (Schmid, *Ber.*, 1886, xix., 1753), but differs from it in percentage composition. Its decomposition with acid would be closely expressed by the equation $C_{36}H_{30}O_{16} + 2H_2O = 2C_{15}H_{10}O_6 + C_6H_{14}O_6$, if rhamnose or glucose are liberated by this reaction. Gallic acid was also isolated, evidently as a decomposition product of gallotannic acid contained in the wood.

Berberis ortuensis, a plant resembling *Berberis vulgaris*, flourishes in Cyprus. It was found to contain berberine, but no colouring matter of the mordant yellow class.

The perianths surrounding the seeds of *Rumex obtusifolius* contain a trace of quercetin, which is interesting, as in many roots of this species methylantraquinone derivatives also exist. It is also pointed out that the leaves and green stems of madder (*Rubia tinctoria*) contain a yellow colouring matter which will be examined.

115. Naphthylureas." By GEORGE YOUNG, Ph.D., and ERNEST CLARK.

The mononaphthylureas may be prepared by the action of potassium cyanate on the hydrochloride of the corresponding naphthylamine. In consequence of the rapid conversion of the mononaphthylureas into the symmetrical dinaphthylureas which takes place on heating, even below the melting-points of the former, the true melting-points have escaped the observation of previous authors. α -Naphthylurea melts at $213-214^\circ$, at which temperature it is converted into di- α -naphthylurea, melting at $284-286^\circ$. β -Naphthylurea melts at $213-215^\circ$, and immediately forms di- β -naphthylurea, melting at $289-290^\circ$. Acetyl- α -naphthylurea, m. p. $214-215^\circ$; benzoyl- α -naphthylurea, m. p. $243-243.5^\circ$; acetyl- β -naphthylurea, m. p. $202-203.5^\circ$; benzoyl- β -naphthylurea m. p. $219-220^\circ$.

116. "Benzoylphenylsemicarbazide." Preliminary Notice. By GEORGE YOUNG, Ph.D., and HENRY ANNABLE.

In a previous communication presented to the Society (*Trans.*, 1897, lxxi., 200), attention was drawn to the disagreement between the melting-points of benzoylphenylsemicarbazide, $202-203^\circ$, as observed by Michaelis and Schmidt (*Ber.*, 1887, xx., 1713), and $210-211^\circ$ as observed by Widman (*Ber.*, 1893, xxvi., 945). It described the preparation and examination of this substance—melting at $202-203^\circ$ —and suggested the possible existence of two benzoylphenylsemicarbazides both having the constitutional formula $C_6H_5N(COC_6H_5) \cdot NH \cdot CO \cdot NH_2$. Shortly after the publication of this paper, Dr. Widman had the courtesy to submit a sample of his preparation for comparison. This sample had been observed by Dr. Widman to melt at $210-212^\circ$; the authors found it to melt at $211-212^\circ$. Their thermometer agreed therefore with Dr. Widman's. A comparison of the properties of the two preparations led to exceedingly interesting results. Widman's benzoylphenylsemicarbazide seemed to be almost, if not quite, insoluble in boiling benzene, the melting-point remaining unaffected. The author's benzoylphenylsemicarbazide was fairly soluble in boiling benzene, crystallising out again on cooling. Mere re-

crystallisation from benzene did not affect the substance, but prolonged boiling with benzene caused a gradual rise of the melting-point. On the other hand, the substance was easily soluble in boiling water and crystallised out on cooling unchanged, whereas Dr. Widman's preparation dissolved in boiling water with difficulty and crystallised out on cooling, with the melting-point considerably lowered. These results induced the authors to undertake a thorough investigation of the formation and properties of benzoylphenylsemicarbazide. They have been able to determine that the action of benzoyl chloride on phenylsemicarbazide produces under different conditions three distinct forms of benzoylphenylsemicarbazide. These three forms melt respectively at $202-203^{\circ}$, $205-206^{\circ}$, and $210-211^{\circ}$. They are each capable of conversion into either of the other two. They exhibit different and characteristic crystalline structures under the microscope. They possess different solubilities and densities. The form of highest melting-point seems incapable of solution without undergoing at least partial change into one or other of the lower melting forms, but pure solutions of these latter may be easily prepared. These solutions have no action on polarised light. The authors are at present engaged in examining the physical properties of these substances and in extending the investigation to a number of other closely related compounds, in the hope of being able to determine whether they are capable of existence in two or more modifications.

117. "Sulphocamphylic Acid." By W. H. PERKIN, jun.

In a previous communication (*Proc.*, 1895, xi., 23) it was shown that when the potassium salt of sulphocamphylic acid is treated with phosphorus pentabromide, the sulphobromide, $C_8H_{12}(SO_2Br)CO_2H$, is produced, and from this substance by elimination of sulphur dioxide an acid of the formula $C_8H_{12}BrCO_2H$ was obtained, which, as it gives β -camphylic acid, $C_8H_{11}CO_2H$, on treatment with alcoholic potash, may be called *bromo-dihydro- β -camphylic acid*.

During the course of further experiments, the corresponding *camphylic sulphochloride*, $C_8H_{12}(SO_2Cl)CO_2H$, has been obtained by treating the potassium salt of sulphocamphylic acid at 0° with phosphorus pentachloride. This substance melts at $168-170^{\circ}$, and at the same time slowly undergoes decomposition with evolution of sulphur dioxide and formation of *chloridihydro- β -camphylic acid*, a crystalline substance which melts at $105-106^{\circ}$.

Like the corresponding bromo-compound, it is decomposed by boiling with alcoholic potash, with elimination of hydrogen chloride and formation of β -camphylic acid.

In the last communication on sulphocamphylic acid (*Proc.*, 1896, xii., 189) it was stated that, when β -camphylic acid was treated with phosphorus trichloride, and the product distilled under reduced pressure, the chloride of an acid melting at 130° is obtained which was called *iso- β -camphylic acid*, because at the time it was thought that this acid might prove to be isomeric with β -camphylic acid. It has since been found that the reaction does not proceed in this way, but that the following much more remarkable change takes place. When the chloride of β -camphylic acid is distilled, there is, as already mentioned, some decomposition and charring, and during this distillation the chloride is *reduced* almost completely to the chloride of an acid, $C_8H_{14}O_2$, which on investigation has been found to be identical with *isolaunonic acid*, the acid which Koenigs and Hoerlin (*Ber.*, 1893, xxvi., 813), and the author (*Proc.*, 1893, ix., 109) obtained by the elimination of sulphuric acid from sulphocamphylic acid.

This same *isolaunonic acid* (together with a liquid acid, which is possibly an isomeride) is obtained when β -camphylic acid is reduced with sodium amalgam under certain conditions, and quite lately it has also been obtained in large quantity by fusing sulphocamphylic acid with soda in a cast-iron pot.

When fused in a nickel dish with caustic soda, sulpho-

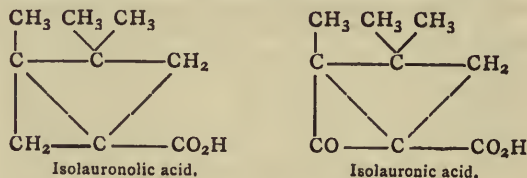
camphylic acid yields a mixture of α - and β -camphylic acids, $C_9H_{12}O_2$, but when an iron pot is used, the iron acts as a reducing agent, and the product, which is found to contain quantities of ferric oxide, on treatment in the usual way yields large quantities of *isolaunonic acid*, $C_8H_{14}O_2$.

Isolaunonic acid is, as Koenigs and Meyer (*Ber.*, 1894, xxvii., 3466) showed, readily oxidised to *isolaunonic acid*, $C_9H_{12}O_3$, a ketonic acid which gives a well characterised oxime and a semicarbazide. On reduction with sodium amalgam, the author finds that *isolaunonic acid* is readily converted into *dihydroisolaunonic acid*, $C_9H_{14}O_3$ (m. p. 88°), a result differing somewhat from that of Koenigs and Meyer, who obtained in this way a lactone, $C_9H_{14}O_2$, melting at $47-50^{\circ}$, together with a substance melting at $80-81^{\circ}$, which they consider to be a mixture of two acids, $C_9H_{14}O_3$ and $C_9H_{16}O_3$.

The author has further studied the action of oxidising agents on *isolaunonic acid*, and finds that, under certain conditions, this acid is split up into dimethylsuccinic acid, $COOH \cdot C(CH_3)_2 \cdot CH_2 \cdot COOH$ and a ketonic acid, $C_8H_{14}O_3$, which melts at 51° .

This ketonic acid on oxidation is converted into *aa-dimethylglutaric acid*, $CO_2H \cdot C(CH_3)_2 \cdot CH_2 \cdot CH_2 \cdot CO_2H$, and it therefore evidently has the constitution $CH_3 \cdot CO \cdot C(CH_3)_2 \cdot CH_2 \cdot CH_2 \cdot CO_2H$, and is identical with the acid previously obtained (*Proc.*, 1896, xii., 190), by oxidising β -camphylic acid.

A careful study of the results obtained in this long series of experiments on sulphocamphylic acid and the acids derived from it, seems to the author to clearly indicate that the constitutions of *isolaunonic* and *isolaunonic* acids are most probably represented by the following formulæ:—



As sulphocamphylic acid on heating is resolved into *isolaunonic acid* and sulphuric acid, and on the other hand, *isolaunonic acid*, as was indicated in a previous communication (*Proc.*, 1893, ix., 109) and has since been proved, when heated with sulphuric acid at 90° , is again converted into sulphocamphylic acid, it follows that the determination of the constitution of *isolaunonic acid* will throw most important light on the formula of sulphocamphylic acid, and on the remarkable changes which take place during the formation of this sulpho-acid from camphoric acid.

The discussion of these points and of their bearing on the constitution of camphoric acid, the author must reserve for a detailed description of his experiments, which he hopes soon to be able to lay before the Society.

ROYAL INSTITUTION.

General Monthly Meeting, December 6th, 1897.

Sir JAMES CRICHTON-BROWNE, M.D., F.R.S., Treasurer and Vice-President, in the Chair.

THE following were elected Members:—The Hon. H. M. Birdwood, C.S.I.; Major John Leslie; Capt. H. G. Lyons, R.E.; and Mr. Cecil Powney.

The special thanks of the Members were returned to Professor Dewar, LL.D., F.R.S., for his present of a portrait of Mr. Benjamin Vincent, Honorary Librarian of the Royal Institution.

NOTICES OF BOOKS.

Handbook (A) for Chemists of Beet-sugar Houses and Seed-culture Farms. Containing Selected Methods of Analysis, Sugar-house Control, Reference Tables, &c. By GUILFORD L. SPENCER, D.Sc., of the U.S. Department of Agriculture. New York: John Wiley and Sons. London: Chapman and Hall, Lim. 1897. Pp. x.—475. 18mo., Limp Morocco Tuck.

It is interesting to note that such great advance has been made in the beet-sugar industry in the United States that already there is an opening for a book devoted exclusively to the sugar-beet. Less than ten years ago, when Dr. Spencer published his "Handbook for Sugar Manufacturers," the manufacture of sugar was confined to that from cane cultivated in the Southern States; sorghum was attracting some attention, but had been nearly abandoned. At present the beet-sugar industry bids fair to attain enormous proportions in America.

The "Handbook" contains, in a compact and attractive form, a fund of information on the chemistry and treatment of sugar-beets that cannot be found elsewhere without access to an extensive library. It treats in sections of sugar-house control; sugar-analysis by optical methods and by chemical methods; sampling and averaging, analysis of the beet, juice, syrup, and molasses, filter press-cake, and residues of divers kinds; analysis of saccharates, bone-black, chimney-gases, as well as of the crude materials used, such as limestone, sulphur, coke, and water; seed selection, seed testing, and special reagents have their share of space. Seventy pages of useful tables are followed by one hundred and twenty-five pages of blank forms for practical use in sugar-house work, besides thirty pages for reporting yield and losses.

The illustrations illustrate the text, and the type, though small, is very clear. Full-face type heads each paragraph.

The volume contains an index.

H. C. B.

CORRESPONDENCE.

CONVERSION OF THERMOMETRIC SCALES.

To the Editor of the Chemical News.

SIR,—In the *Meteorologische Zeitschrift* for the month of October, G. Hellmann (see also *Revue Scientifique*, November 20, 1897, p. 663), there is an ingenious formula for the conversion of Fahrenheit degrees into those of Centigrade, which may interest you if you have not seen it. The usual formula is, of course,—

$$\frac{(F^{\circ} - 32) \times 5}{9} = C^{\circ}.$$

G. Hellmann:—

$$\frac{5}{9} = 0.5555 = \frac{1.111}{2} = \frac{1}{2} + \frac{0.1}{2} + \frac{0.01}{2} + \dots$$

Let 88 be the number of degrees Fahrenheit, we have then—

$$\begin{aligned} 88 - 32 &= \frac{56}{2} = 28. \\ &\quad 28 \\ &\quad 2.8 \\ &\quad 0.28 \\ \hline &31.08 C^{\circ} \end{aligned}$$

I would venture to point out that Reamur's scale may be converted into that of Celsius in a somewhat similar manner.

$$\frac{4}{9} = 0.4444 \times 2.5 = 1.0 = 2.5 \times 4 = 1.0, \\ \left(\frac{F - 32}{10} \right) \times 4 = R^{\circ}.$$

Example:—

$$\begin{aligned} \frac{212 F - 32}{10} &= 18 \times 4 = 72.00 \\ &\quad 7.20 \\ &\quad 0.72 \\ &\quad 0.07 \\ \hline &= 79.99 \end{aligned}$$

While the conversion of Celsius to Fahrenheit is given as follows:—

$$\frac{9}{5} = 0.180 \times 5.555 = 1 = 0.5555 \times 2 = 1,$$

we have then—

$$32 - \left(\frac{C^{\circ} \times 2}{10} \right) + C^{\circ} \times 2 = F^{\circ}.$$

Example:—

$$32 - \frac{100 C \times 2}{10} = 32 - 20 + 200 = 212^{\circ} F.$$

The advantage which these formulæ present is, as shown by M. Hellmann, that these conversions may be conducted mentally.—I am, &c.,

THOS. PALMER.

4, Rue Haringrode, Antwerp,
November 27, 1897.

ON NEW SPECTRAL LINES OF OXYGEN.

To the Editor of the Chemical News.

SIR,—In the abstracts from foreign journals in the *CHEMICAL NEWS* of November 26 (vol. lxxvi., p. 265), you were good enough to notice my recent discovery of new lines in the spectrum of oxygen, but the wave-lengths, as set forth in the *Comptes Rendus*, have not been given. May I ask you to kindly publish the enclosed copy of a letter from Dr. Schuster, addressed to me in May last, which supplies the omission, and at the same time establishes—by the date—my independent discovery of these lines. The diagrams of the spectrum of oxygen published by Profs. Runge and Paschen in *Wiedemann's Annalen* of July last, and reproduced in the *CHEMICAL NEWS* of November 26th, shows two lines in about the same positions as those which I have observed.—I am, &c.,

H. WILDE.

The Manchester Literary and Philosophical Society,
36, George Street, Manchester, Dec. 7, 1897.

[COPY].

4, Anson Road, Victoria Park,
May 7th, 1897.

DEAR MR. WILDE,

I saw to-day your two new oxygen lines, and asked Mr. Hensalech to measure them so as to make sure they were the same. He finds the wave-lengths 7759 and 7165, agreeing well with yours (7760 and 7160); for with the small spectroscopic we had to-day we could not be sure to a few units of these numbers. The lines Mr. Hensalech saw previously must have been nitrogen lines.

Yours sincerely,
(Signed) ARTHUR SCHUSTER.

SUGAR-BEET.

To the Editor of the Chemical News.

SIR,—I shall be glad if you will announce in your next issue that I am distributing sugar-beetroot seed gratuitously to anyone willing to grow sugar-beetroots experi-

mentally next season, and that I will analyse the roots and report thereon free.

The consumption of sugar in the United Kingdom amounts to over one million tons per annum, representing a value of £19,000,000, which all goes to the foreign exporter, and which should be kept in this country if possible.

I was closely connected with the experiments of sugar-beetroot growing carried on in this country about two or three years ago, and by which it was proved that the roots can be grown here quite as well, if not better, than on the Continent, by such well-known landed proprietors as Lord Rosebery, Lord Winchilsea, and Lord Jersey.

Great Britain, with its large consumption of 78 lbs. per head per annum, should grow its own sugar, and thus prevent the closing of any more sugar-refineries, and also be the means of re-opening the large factories now closed in London, Liverpool, Glasgow, Greenock, Bristol, Plymouth, Manchester, and Dublin.

If gentlemen will send me their names and addresses I will forward the seed to them in time for next spring's sowing, and I shall have much pleasure in aiding, gratis, in the formation of any syndicate to erect factories to manufacture the sugar.—Thanking you in anticipation, I am, &c.,

SIGMUND STEIN.

323, Vauxhall Road, Liverpool,
December 1, 1897.

MISCELLANEOUS.

Royal Institution.—The following are the Lecture Arrangements before Easter:—Professor Oliver Lodge, six Christmas Lectures (specially adapted for young people) on "The Principles of the Electric Telegraph"; Professor E. Ray Lankester, eleven lectures on "The Simplest Living Things"; Professor Dewar, three lectures on "The Halogen Group of Elements"; Dr. J. Paul Richter, three lectures on "Some Italian Pictures at the National Gallery"; Professor J. A. Fleming, five lectures on "Recent Researches in Magnetism and Diamagnetism"; Professor Patrick Geddes, three lectures on "Cyprus"; Mr. Wm. H. Hadow, three lectures on "The Structure of Instrumental Music"; Mr. Lionel Cust, two lectures on "Portraits as Historical Documents, Portraits as Monuments." The Friday Evening Meetings will begin on January 21st, when a Discourse will be given by the Right Hon. Sir John Lubbock, Bart., M.P., on "Buds and Stipules"; succeeding Discourses will probably be given by Professor C. Lloyd Morgan, Mr. A. A. Campbell Swinton, Dr. J. Hall Gladstone, Professor L. C. Miall, Captain Abney, Professor J. E. Thorpe, Mr. James Mansergh, the Dean of Canterbury, Professor Dewar, and other gentlemen. Lord Rayleigh will deliver lectures after Easter.

London County Council.—An interesting ceremony took place on Saturday, November 20th, at the Holborn Restaurant, when the members of the staff of the Chemical and Gas Department of the London County Council gave a complimentary dinner to their late chief, Mr. W. J. Dibdin. The new chief of the department, Dr. F. Clowes, of Nottingham College, took the chair, and in proposing the toast of the evening, pointed out that the work carried out by Mr. Dibdin as chief of the department reflected credit alike on the Council and himself. In conclusion, the Chairman presented to Mr. Dibdin, on behalf of the officers of the department, an illuminated address. Mr. Livingston, senior officer of the department, who read the Address to the company, bore testimony to the invariable kindness and courtesy of their late chief, and, referring to his public usefulness, gave as an example his successful treatment of the London sewage. The next

toast, "The Chairman," was proposed by Dr. Teed in an appropriate and somewhat amusing speech. In replying to which Dr. Clowes confided to the company the "nickname" by which he is known among his students at Nottingham, and which promises well for the continuance of the good feeling between chief and officers that has heretofore existed. On account of the recent arduous task imposed upon a sister Department, the toast "The Fire Brigade," proposed by Mr. Livingston, was enthusiastically received. The evening's proceedings were further enlivened by the music provided by Messrs. J. W. Heath and F. Goddard, professionally assisted by Mr. L. Thorne, who artistically rendered "Mary of Argyle," &c., and by Mr. J. W. Kipps, F.R.C.O., A.R.A.M., whose performance at the piano was greatly appreciated. Mr. Edward Minshall favoured the company with two excellent recitations, entitled "The Benediction" and "My First and Last Cricket Match." A vote of thanks to the Hon. Sec., Mr. E. J. Jackman, terminated the proceedings.

Deterioration of Paper.—Sir H. Trueman Wood writes to the *Athenæum*:—A committee of the Society of Arts is now at work on the subject of the deterioration of modern paper. It is a matter of general repute that many books are now printed on paper of so inferior a character that it is liable to perish in a short space of time; but the committee are anxious to have definite examples before them of books that have thus suffered. Might I ask if any of your readers who have had experience of such cases would kindly communicate the facts to me; and also if they would send me any examples of books printed within the last fifty years in which the paper shows signs of perishing? I need not say that any such books will be carefully preserved, and, after the committee have had an opportunity of inspecting them, returned to the lenders."

On the Determination of Silica in Blast Furnace Slag.—G. H. Meeker (*Y. Am. Chem. Soc.*).—The dehydration of the silicic acid by means of concentrated sulphuric acid, instead of by heat, is the essential feature of the procedure, for which both rapidity and accuracy are claimed. The filtrate from the silica thus obtained cannot, however, be utilised for the determination of either aluminum or calcium. The method yields satisfactory results in the presence of spinel.

MEETINGS FOR THE WEEK.

MONDAY, 13th.—Society of Arts, 8. (Cantor Lectures). "Gutta Serena," by Eugene F. A. Obach, Ph.D., F.R.S.

WEDNESDAY, 15th.—Society of Arts, 8. "The Purification of Sewage by Bacteria," by Samuel Rideal, D.Sc.

— Microscopical, 8. "A New Form of Photomicrographic Camera and Condensing System," by E. B. Stringer, B.A.

— Institution of Mining and Metallurgy, 8. "Mining on the Black Reef, Witwatersrand Goldfields, South Africa," by W. Fischer Wilkinson. "Notes on Smelting at Broken Hill," by Henry Watson. "Notes on the Buying and Sampling of Ores, and the Working of Mines on the Tribute System, in Chili," by Gerald V. Hopkins.

— Chemical, 8. (Extra Meeting). "Kekulé Memorial Lecture," by F. R. Japp, LL.D., F.R.S.

THURSDAY, 16th.—Chemical, 8. "Stereo-chemistry of Unsaturated Compounds—Part I., Esterification of Substituted Acrylic Acids," by J. J. Sudborough, Ph.D., and Lorenzo Lloyd. "Formation and Hydrolysis of Esters," by J. J. Sudborough, Ph.D., and M. E. Feilmann, B.Sc. "A New Method of Determining Freezing-points in very Dilute Solutions," by M. Wilderman, Ph.D.

NOTES AND QUERIES.

* * Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Penetration of X Rays.—Could anyone inform me (per CHEMICAL NEWS) in what ratio the X rays can penetrate plates of equal thickness of the different metallic elements as regards their atomic weights. Thus, the atomic weight of Fe is 56 and Al 27; seeing that the atomic weight of one is practically double the atomic weight of the other, would Fe offer twice as much resistance to the rays as Al would, and, if not, is there any ratio?—X. Y. Z.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1986.

THE ESTIMATION OF COPPER IN PRESENCE OF OTHER ELEMENTS.

By HARRY BREARLEY.

In a previous paper (CHEMICAL NEWS, vol. lxxvi., p. 189) it was shown that the claim to increased accuracy brought about by the substitution of soda carbonate and soda salts for ammonia and its salts in the cyanometric estimation of copper was a just one. It was also pointed out that the silver iodide end-reaction, which has already been used in the titration of nickel, cobalt, and mercury solutions, could be made supplementary to the colour indicator, or, if need be, act alone.

Two lengthy tables setting forth the influence of certain associated elements on the estimation of copper by cyanide, when performed in the usual way, have already appeared in the CHEMICAL NEWS (Field, i., 61; Thomson, xxxiii., 152), while a writer here and there has chronicled the quantitative behaviour of one or two elements.

It is proposed now to state the observed interference of a similar series of elements, using the AgI turbidity as indicator. The advantage of such an indicator in eliminating the personal error, especially when a precipitate forms in the solution, has been elsewhere commented on.

It must not be presumed that the writer attempts to decide between the differences of previous observers, although the results are not without value in this respect. The aim is to show that in every variety of circumstance the new method of operating is better than the old; the comparisons of Table I. are made to that end.

The new mode of operating is found to be especially serviceable where the associated element is precipitated in the titrated liquid. Very few of such precipitates are entirely without influence on the colour indicator, as Thomson's results show, while others so completely mask it as to make any reliable observations altogether impossible until the precipitate has been allowed to settle. This delay in a process which depends in any degree on the rate at which the reagent is added cannot be satisfactory. Nor can a separation at any stage of the process always overcome this difficulty, on account of the tenacity with which ammoniacal copper solutions adhere to many precipitates, even when the separation chances to be complete.

The observations are summarised in Table I. The following are details common to each test:—Amount of copper, 0.1000 grm.; alkali salts, 20 c.c. HCl (2 normal strength), as chloride; excess of soda carbonate, 30 c.c. (2 N); excess of ammonia, 10 c.c. (2 N). A decreased excess of ammonia was used so as to quite avoid the interchange of silver and copper. Under the head of "Percentage Interferences" are those found in this investigation and those found by Field and Thomson. The soda carbonate series explains itself.

Where the registered interference is below $\frac{1}{4}$ per cent, it may be taken that the element is really without influence, and that such small interferences are due to experimental error. It must be understood that the results stated were obtained by operating in the manner explained in the previous paper. Where the interference is considerable, such attempts as have been made to obviate it, with the measure of success, are stated in the next section.

The behaviour of a method in the hands of different operators is a good index of its value. The ordinary cyanide titration is much better than a comparison of the results of Thomson and Field might suggest. But such

a comparison does show how very susceptible the ammoniacal cyanide procedure is to more or less of what it is desirable should be, in the final operation, inactive reagents.

The Behaviour of the Added Elements. Means of Minimising some Interferences.

The most obvious means of overcoming an interference is to separate the offending element before estimating the copper. Such means need not be further referred to. It is intended here to show how, by modifying the titration, and without any separation whatever in a quantitative sense, the copper may be estimated with sufficient exactness to meet technical demands.

Where the form in which the element was added is not stated, it may be taken that the chloride was used.

Sodium has frequently been shown to be without influence.

Potassium may be presumed to be similarly inactive.

Calcium.—Ammonia, no precipitate. Soda, precipitate easily filterable. The precipitate seems to hold the colour somewhat even when the solution is decolorised. A greater excess of cyanide is therefore advisable. Thomson explains his high result by presuming that there is some reaction between the cyanide and the calcium salts. His cyanide contained considerable quantities of carbonate. May not the adherent colour more truly account for the error?

Barium and Strontium.—Similar remarks apply to these metals.

Magnesium.—No precipitate in either case.

Zinc.—More than usual interest attaches to this element, because the alloys of copper and zinc are so abundant. On this account the greatest pains have been taken to minimise its influence. Under this head will be noticed a number of modifications which may be applied to other elements.

Zinc-Ammonia.—An excess of 10 c.c. 2 normal ammonia is not sufficient to precipitate the zinc and re-dissolve it; 30 c.c. was used. The most noticeable feature of titration is the large excess of cyanide, over and above that actually required to combine with the zinc, needed to discharge the colour.

Zinc-Soda.—On making alkaline with soda carbonate the zinc is of course precipitated. On adding the cyanide the colour goes as usual, until the theoretical amount of cyanide is added. It is possible at this point to get a colourless solution, by prolonged standing, holding the greater part of the zinc precipitated; but the precipitate is not colourless. It remains a pale green in the most persistent manner until enough cyanide has been added to dissolve it completely, or nearly so. The excess of cyanide required to do this when copper and zinc were together in equal amounts varied from 60 to 80 per cent more—according to volume, time of standing, &c.—than was needed to react with the copper alone.

On adding the potassium iodide, and then the silver nitrate, there appeared a white precipitate, in many ways unlike the creamy silver iodide. It was filtered off, more AgNO₃ added, and again the white precipitate appeared, and so on the precipitation and filtration might alternate until the silver iodide turbidity did appear.

This at once suggests that the zinc which had combined with the cyanide is displaced by the silver, and that the repeated precipitations and filtrations are unnecessary if enough silver nitrate is at once added to replace the whole of the zinc.

In doing this it was possible to use the potassium iodide to indicate when enough silver had been added. It is not difficult to observe this point when as much as 0.1 grm. of zinc is present, because the zinc carbonate, previously translucent and white, becomes, owing to admixed AgI, opaque and yellowish, and, instead of settling in flakes, remains suspended in no determinate shape. But the indication is hardly delicate enough to accept as the end of the operation, and so a little cyanide is added, which

TABLE I.

METAL.	AMMONIA SERIES.						SODA CARBONATE SERIES.			
	Copper found.			Percentage error.			Copper found.			Percentage error.
Grm. added ..	0.01	0.05	0.10	H. B.	Thomson.	Field.	0.01	0.05	0.10	
Calcium ..	0.1000	0.1000	0.1005	0.5	1.35	0.0	0.1000	0.0997	0.1000	0.0
Barium ..	—	0.1000	0.0996	0.4	1.90	0.0	0.1000	0.1002	0.1000	0.0
Strontium ..	—	0.0998	0.1000	0.0	2.55	0.0	—	0.1002	0.1000	0.0
Magnesium ..	—	0.1004	0.1002	0.2	0.90	0.0	—	0.0998	0.1002	0.2
Zinc ..	1012	0.1054	0.1094	9.4	20.65	25.0	—	0.1015	0.1020	2.0
						34.6*				
Cadmium ..	—	0.1010	0.1028	2.8	6.85	—	—	0.1004	0.1000	0.0
						15.4*				
Aluminium ..	0.1003	0.1006	0.1007	0.7	3.55	0.0	0.1006	0.1007	0.1007	0.7
Iron ..	0.1002	0.1002	0.0998	0.2	0.85	0.0	0.1000	0.0994	0.0987	1.3
Manganese ..	0.0968	0.0964	0.0969	3.1	24.85	12.0	0.0977	0.0966	0.0970	3.0
Chromic oxide ..	—	0.0869	0.0730	27.0	—	0.0	0.0992	—	0.0975	2.5
Chromic acid ..	—	—	—	—	—	—	—	0.1002	0.0996	0.4
Tin ..	—	0.1000	0.0998	0.2	1.35	0.0	—	0.1000	0.1003	0.3
Molybdenum ..	—	0.0998	0.1000	0.0	0.15	—	—	0.1004	0.1002	0.2
Arsenic ..	—	0.0998	0.0998	0.2	0.0	0.0	—	0.0998	0.0998	0.2
Antimony ..	—	0.1000	0.1004	0.4	1.9	0.0	—	0.1002	0.1004	0.4
Bismuth ..	—	—	—	—	2.45	0.0	—	0.1002	0.1000	0.0
Lead ..	—	0.1002	0.1000	0.0	0.4	0.0	—	0.0998	0.1002	0.2
Mercury (ous) ..	—	0.1062	0.1120	12.0	19.1	—	—	1070	113.9	13.9
„ (ic) ..	—	0.1089	1182	18.2	23.2	50.0	0.1018	0.1107	0.1200	20.0
Uranium ..	—	0.1004	0.1004	0.4	2.45	—	—	0.0998	0.1002	0.2

* Dulin, *Journ. Amer. Chem. Soc.*, May, 1895.

dissolves the AgI without dissolving more than traces of the zinc. The solution is then filtered and finished in the ordinary way. An alternative is to add an excess of silver nitrate, but no potassium iodide, filter, and titrate the excess with cyanide. The soda results in Table I. were obtained in this way.

Such a procedure as the above even apart from the error cannot be widely serviceable, on account of the expense. An attempt was made to substitute nickel for silver, for economy's sake, and with the idea that the error on using silver was possibly due to its inability to completely displace the zinc. The fear was that nickel, being more active to cyanide than silver, might also displace the copper. This, however, it did not do, but being only partially soluble in soda carbonate it did necessitate an examination of the residue, as well as the filtrate, before the excess could be found. The multiplicity of operations pending such a procedure led to its rejection.

One point, which ought never to have been overlooked, was strongly emphasised; that is, the nickel may be associated with the zinc as carbonate in a solution which contains an excess of free cyanide. This recalls the tenacity with which the copper was retained in the zinc carbonate, and suggests that an examination of the residue might have revealed the amount of silver necessary to correct the 2 per cent error.

Other reagents, such as tartaric and citric acids, were tried. They did not eliminate the error. They are objectionable, too, for reasons stated below.

The most satisfactory modification lies in the use of sodium pyrophosphate (prepared by heating Na_2HPO_4 to redness). A saturated solution was used. Its influence on the titration of pure copper solutions is shown below:—

0.0	25.0	50 c.c. pyrophosphate.
0.0500	0.0504	0.0502 gr. Cu indicated.

This reagent was suggested by T. Moore (*CHEM. NEWS*, lix., 160), for titrating nickel in presence of zinc. Its only defect is a slight lagging in the reaction between the cyanide and silver iodide which accompanies its use.

This lagging may be counterbalanced by adding the same volume of pyrophosphate to the standard copper. Two results obtained in this way are:—

0.0	0.05	0.1 gr. zinc present.
0.1000	0.1008	0.1006 gr. copper found.

In other hands it has given as good and better results.

Cadmium.—The reactions very much resemble zinc, but of much less intensity. The precipitate formed on making alkaline is in each case dissolved by the cyanide. With sodium carbonate the cadmium was re-precipitated on adding the silver nitrate, as explained above, and as no great excess of cyanide is needed, and the titration offers no other difficulty, there was no need to try further modifications, although doubtless the pyrophosphate would answer with cadmium as well as with zinc.

Aluminium.—There is a precipitate with either alkali. These precipitates are objectionable in that they are difficult to filter off, and the prolonged exposure of even dilute cyanide solutions tends to give high results.

The difficulty, and a similar one with other elements, may be overcome in several ways:—

I. Fractional filtration; but great care must be taken that the fraction preserves its proportionate volume throughout the succeeding operations. It is a good plan to fractionate the standard similarly.

II. The bulk of silver nitrate necessary to combine with the free cyanide may be added before filtration. This precaution may also be taken along with fractional filtration. It minimises the effect of prolonged exposure, and may be safely used for any of the elements (except chromium) in Table I., without fear of the objectionable features accompanying zinc carbonate.

III. The formation of precipitate may be prevented. This can be done by adding tartaric or citric acid, soda pyrophosphate, or making alkaline with soda or potassium hydrate. The two acids so generally employed for similar purposes cannot be recommended here. So small a quantity, about 1 grm. of tartaric acid (say), as suffices to keep 0.1 grm. of aluminium in solution has a very objectionable influence on the titration. The colour of the alkaline solution is less deep than without the acid, and as the proportion of aluminium rises the later colourations on adding cyanide shade off into pale green, and are of little use as indicators; but the most objectionable feature is the way in which a turbidity once formed gradually disappears, so that one is left at the mercy of all manner of evil suggestions, however conscientiously the work may

be done. It may be possible to wait for a disappearing turbidity, and replace, and so on, until all errors vanished, but such a procedure is not practicable. It is this defect, in a much smaller degree, which was referred to under zinc as accompanying pyrophosphate. Citric acid behaves similarly. These remarks are based on an observance of soda solutions only.

Seven or eight of the tabulated elements give no precipitate when soda carbonate is replaced by soda hydrate. The behaviour of such solutions may be mentioned, but the procedure can be recommended only within narrow limits. There should not be a greater excess than 10 c.c. 2 normal NaHO in about 250 c.c. of solution, else the displacement of Cu by silver takes place; and the standard should be worked alongside the sample, because the turbidity after standing awhile becomes brown.

(To be continued).

ZINC IN WATER.

By PERCY A. E. RICHARDS, F.I.C.

As the contamination of water by zinc is not of very frequent occurrence, perhaps the following instance may be of interest:—

The water was drawn from a Berkshire district, and, after being stored in a reservoir, was supplied to a private residence for drinking purposes, by a galvanised iron pipe about 2 miles in length.

When first received the sample was perfectly bright and clear, but when left exposed to the air for about an hour it developed a distinct scum on the surface.

The following results were obtained from an analysis of the water:—

Free ammonia	0.0042	grain per gallon.
Albumenoid ammonia..	0.0028	" "
Nitrogen as nitrates ..	0.035	" "
Chlorine as chlorides ..	1.3	grains "
Total solid constituents	14.35	" "
Zinc bicarbonate	5.12	" "
Iron	traces.	" "

The presence of the zinc was easily detected in the unconcentrated water by both the ammonium sulphide and potassium ferrocyanide tests. Upon boiling the water a precipitation of carbonate of zinc took place.

The "total hardness" of the water drawn direct from the reservoir was equivalent to 4.5 grains of calcium carbonate, and the total solid constituents were only 10 grs. per gallon.

ESTIMATION OF CHLORINE, BROMINE, AND IODINE IN SALINE WATERS.

By PERCY A. E. RICHARDS, F.I.C.

VARIOUS methods have been suggested at different times for the separation of the halogens from mixtures of the three. For instance, Donath has propounded a process based on distillation with chromic acid, as a means for the separation of bromine.

Again, Vortmann recommends boiling a known volume of the solution containing the three halogens, in the first place with pure manganese dioxide and acetic acid. This causes the liberation of the whole of the iodine, and this can then be boiled off. The chloride and bromide in the residue may next be estimated in terms of decinormal silver nitrate solution, when the difference between this number and the equivalent in silver nitrate for the same volume of the untreated water gives the amount of decinormal iodine present.

A known volume of the water is next boiled with per-

oxide of lead and acetic acid, and the bromine and iodine thus liberated boiled off. The silver nitrate equivalent of the residue is determined as before, and the bromine calculated by difference between this number and the previous determination.

These processes give satisfactory results so long as one of the halogens is not present in much greater amount than either of the other two. But in the examination of waters it is almost invariably the case that the amount of chlorine greatly exceeds that of both bromine and iodine, sometimes to the extent of 1000 or 2000 to 1. In cases like these, the estimation of iodine and bromine by difference is of very little value. I had occasion recently to examine some waters of this character, and found that the following methods gave very good and concordant results:—

In the first place, the total halogen equivalent in terms of decinormal silver nitrate solution was accurately determined. The iodine present was then estimated by treating 250 or 500 c.c. with acetic acid and hydrogen peroxide for about half an hour, and extracting with chloroform. The mixture was shaken in a separating funnel, with successive small quantities of chloroform, and these washings added to the first portion. The solvent was then well washed with distilled water to remove any peroxide of hydrogen, and was then titrated with decinormal sodium thiosulphate solution, and the amount of iodine calculated. This process, which is due to Cook, is simple and accurate.

The bromine was next estimated by shaking the iodine-free liquid left in the separating funnel, with chlorine water and chloroform, avoiding much excess of the former. The solvent was drawn off, and the aqueous solution shaken several times with small quantities of chloroform, the latter being added to the first portion. Any chlorine present is washed out of the chloroform by repeated agitation with distilled water, and the bromine estimated by adding a few crystals of iodide of potassium and titrating with thiosulphate of soda solution.

The iodine and bromine present being now known, the silver nitrate equivalents can be calculated and subtracted from the total halogen equivalent, and from this result the chlorine present is deduced.

This process enables the bromine and iodine to be estimated directly and with considerable accuracy, and is therefore specially suited to the examination of saline waters where the quantities present are relatively small.

A REVISION OF THE ATOMIC WEIGHT OF NICKEL.*

FIRST PAPER.—THE ANALYSIS OF NICKELOUS BROMIDE.

By THEODORE WILLIAM RICHARDS

and

ALLERTON SEWARD CUSHMAN.

(Continued from p. 286).

Purification of Materials.

Nickel.—Since the possibility of preparing and analysing pure nickelous bromide had now been proved, the next step was to purify all the materials concerned in its manufacture. First among these materials comes nickel, which must not only be freed from all known impurities, but must also be so treated as to detect and eliminate unknown ones. With this latter purpose in view, our material was obtained from two distinct sources; first, the "pure" nickel of commerce; and, secondly, really pure nickel (containing only a little iron) prepared by Dr. Mond

* Contribution from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, vol. xxxiii., No. 7.

through the carbonic oxide process and kindly presented by Dr. Wolcott Gibbs.

It is convenient to consider first our treatment of the commercial nickel. In proceeding with the further purification of this sample, our first step was to remove the metals of the copper and tin groups. The simple treatment with hydric sulphide has generally been considered sufficient to insure the separation of the metals of these groups, in spite of the fact that many of the sulphides when present in small quantities often assume a colloidal condition in which they cannot be separated by filtration. In our case this difficulty was avoided by regulating the acidity of the solution so that a certain amount of black nickelous sulphide was precipitated, which effectually "swept" this liquid, coagulating small quantities of foreign sulphides. After filtration the liquid was boiled to drive off the hydric sulphide, oxidised with a few drops of nitric acid, made alkaline with ammonia, and filtered. The precipitation of the sulphide was now continued by the passage of a little washed hydric sulphide. This first comparatively small amount of sulphide was filtered out and discarded. The remaining nickel was then as nearly as possible completely precipitated in the form of sulphide. After the precipitate had been allowed to settle in a closed flask over night, the liquid was decanted, and the precipitate was washed by decantation many times with boiling water until neutral. Cold dilute hydrochloric acid was now added, and the precipitate was digested for several days. Since the colour of the supernatant liquid showed that a slight amount of even the comparatively insoluble nickel sulphide had gone into solution, the assumption was not unreasonable that nearly, if not quite, all of the more soluble sulphides must have been removed. The black precipitate was now repeatedly washed with hot water until the washings were quite neutral; it was next dissolved in strong hot hydrochloric acid, and, after the separated sulphur had been removed, the solution was evaporated to dryness, and the residue was taken up with water. The material was now considered fairly free from its usual impurities, with the single exception of cobalt.

In commenting on the work of an early experimenter upon the atomic weights of nickel and cobalt, Clarke has objected that "his results are entitled to no especial weight at present, since it cannot be certain from any evidence recorded that the oxide of either metal was absolutely free from traces of the other" ("Re-calculation," 1897, p. 291). Since the two metals have atomic weights only differing at the outside by half a unit, "traces" of one in any preparation of the other metal could not alone furnish a reason for invalidating the results. Nevertheless, for our purpose it seemed desirable to prepare nickel as nearly free from cobalt as possible. In order to attain this end with any degree of certainty, it is obvious that a qualitative test must be found that should show with sufficient accuracy the presence or absence of cobalt. Winkler (*Zeit. Anal. Chem.*, vi., 20) has recommended a test for which he claimed greater accuracy than the better known method with potassium nitrite. The moderately dilute solution of nickel is treated with ammonia until a clear blue colour is obtained, and then one or two drops of potassic permanganate are added. If no cobalt is present, the blue solution of nickel takes on a purple tinge; whereas cobalt, if present, reduces the permanganate. Winkler does not state the dilution of the permanganate solution, or how much should be added, although manifestly the degree of refinement of the test depends on these points. The permanganate solution which worked well with us contained 1 grm. of the salt in a litre. To the dilute solution of nickel to be tested, contained in a colour-comparison apparatus, enough ammonia is added to render the solution a light sky-blue, and then one-tenth of a c.c. of the permanganate is dropped in. Under these conditions, the mixture appears decidedly purplish in hue, if cobalt is absent. Of course, the test is of value only in the absence of any foreign substances having either

a reducing or an oxidising action on permanganate. We have found it possible by this method to detect one part of cobalt in 2500 parts of nickel, an amount of impurity which could cause a final error in the atomic weight of only 1 part in 500,000.

Anthons' process (see Dammer, *Anorg. Chem.*, iii., 490) for eliminating cobalt was adopted for the purification of our sample. The nickel was twice fractionally precipitated as hydroxide by means of pure sodic hydroxide, the mixture being thoroughly boiled. As far as our test could show it, all the cobalt remained in the filtrate, the last precipitate being contaminated only with a small amount of alkali.

When ammonia is added to a solution of nickelous bromide, a beautiful violet crystalline compound is formed, having the formula $\text{NiBr}_2 \cdot 6\text{NH}_3$, according to our analysis as well as those of Rammelsberg (*Pogg. Ann.*, lv., 243). Since this compound is characteristic of nickel, and similar compounds are not formed by cobalt or most other metals under similar circumstances, and since it is soluble in strong hot ammonia water, but almost insoluble in cold ammonia, it affords a very convenient and effectual means of purifying nickel preparations. Our purified oxide was hence dissolved in pure hydrobromic acid, ammonia was added in excess, and the mixture contained in a platinum flask, was cooled to zero. The beautiful purple precipitate was collected upon pure filter-paper and was washed with strong ammonia. All the material used in the analyses was passed through this treatment at least once, although the various samples were subsequently subjected to different methods of further treatment which will be described in each case.

The violet compound made from our first sample of purified nickel was treated with an excess of water and boiled in a platinum dish, a proceeding which completely precipitated the nickel as hydroxide. The greenish mass was thoroughly washed, and was then dissolved in hydrobromic acid. The nickelous bromide thus obtained was dried in a vacuum desiccator over dry soda; but even after this treatment it was found to have retained considerable quantities of water, an impurity which greatly increased the difficulties of sublimation. Hence this sample, numbered I., was used only for two preliminary analyses.

Another portion of this same sample of the violet compound was re-crystallised several times in succession by cooling its hot ammoniacal solution. The resulting magnificent crystals were dissolved in water and the solution was boiled to drive off the ammonia. The precipitation being thus accomplished, the basic hydroxide was collected, carefully washed, dried, and ignited over an alcohol flame in a porcelain vessel. The resulting nickel oxide was reduced to the metal by igniting this material, held by a porcelain boat in a combustion-tube through which a current of pure dry ammonia gas was passing. The spongy nickel thus produced was changed to bromide and sublimed in the manner already described. The pure substance thus prepared is designated below as Number II.; it also served for two of the earlier analyses.

The further purification of the sample of nickel obtained by the process of Mond, Langer, and Quincke, at first proceeded exactly in the steps just described; except that no attempt was made to remove cobalt, since none was present. After it had reached the stage of treatment represented by the last hydroxide obtained above, the material was converted into the sulphate and subjected to electrolysis out of an ammoniacal solution in a platinum dish. The object of this procedure was, of course, to free the nickel still more effectually from the alkalis, silica, and many other impurities which are not precipitated on the cathode. The bright heavy deposit of pure nickel was dissolved, with a great deal of difficulty, in re-distilled strong nitric acid; and the excess of acid was driven off by evaporation. Ammonia and a large excess of pure water were now added, and the solution was boiled until the basic hydroxide was completely precipitated. This

was subsequently changed to metallic nickel and then into the sublimed bromide, in the manner already described, and the resulting material, labelled III., served for a large number of analyses. A small portion of it, that used for Experiment 8, was re-sublimed.

The sample of material used for the final analysis was even more carefully purified than this, however. A quantity of the pure nickelous oxide of about the grade of purity of Sample III., coming originally from the Mond nickel, was dissolved in sulphuric acid, and the solution was made alkaline by passing in pure ammonia gas, in a platinum dish. When most of the nickel had been deposited electrolytically from this solution, the portion remaining in the electrolyte was thrown away. The bright coating of nickel was washed, pure dilute sulphuric acid

Thus, while all of our samples of nickelous bromide analysed had been sublimed, the several samples had received previous to sublimation very varying treatment. The fourth had been put through a process of purification far more searching than the first, which had merely been freed from the ordinary known impurities. Hence the essential identity of the results obtained from these several samples is very striking.

Purification of other Materials.—Silver was purified exactly in the manner described by Richards and Parker (*Proc. Amer. Acad.*, xxxii., 62) in a recent paper upon the atomic weight of magnesium. The electrolytic crystals were finally fused upon a boat of pure lime in a vacuum. For further details the above-mentioned paper should be consulted.

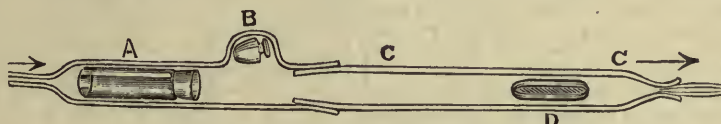


FIG. 2.—BOTTLING APPARATUS, HORIZONTAL SECTION.

A, Weighing bottle. B, Stopper of bottle. C C, Hard glass tube. D, Platinum boat containing nickelous bromide.

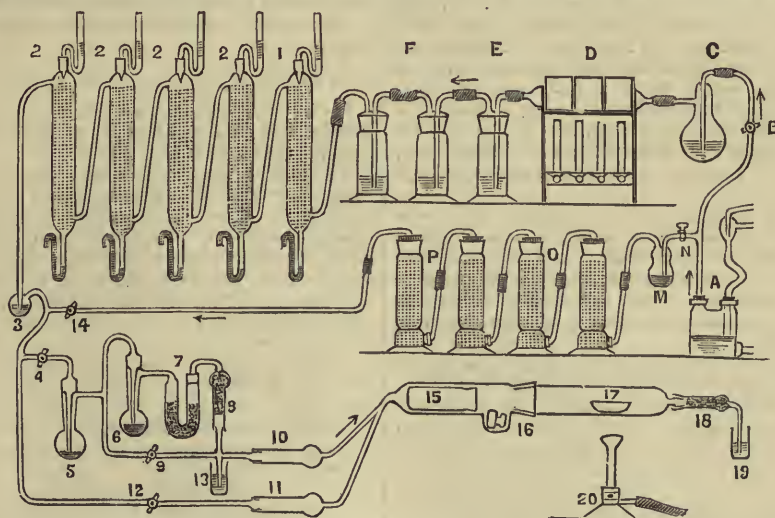


FIG. 3.—APPARATUS FOR IGNITING NICKELOUS BROMIDE IN ANY DESIRED MIXTURE OF GASES.

The use of rubber was confined to the first part of this train, where it could do no harm: (A B C D E F and A M N O P).

was put into the dish, and with reversed poles a strong current was sent through the solution until nearly, but not quite, all of the nickel was dissolved. The solution was then decanted into another dish, ammonia was passed in until the precipitate formed had re-dissolved, the poles were again reversed, and then nickel was once more almost all deposited. This cycle of operations, which gives an excellent method of fractionation, was repeated three times. The final deposit of nickel was dissolved by filling the platinum dish with pure dilute nitric acid and reversing the poles. Only one who has tried dissolving a deposit of nickel on a platinum dish, even in strong nitric acid, can appreciate the ease, cleanliness, and convenience of this method of procedure. The solution of nickel nitrate thus prepared was concentrated by evaporation, and ammonia was passed in until a mass of crystals of the blue ammonio-nickel nitrate was formed. After the mother liquor had been poured off, the crystals were washed with pure ammonia water, and were finally boiled in an excess of pure water in the same platinum dish. The resulting basic hydroxide was then changed to spongy nickel and nickelous bromide in the usual fashion, bearing the title No. IV.

With the co-operation of Mr. Baxter, bromine was purified in a preliminary fashion by solution in strong aqueous calcic bromide, and a subsequent separation. Carefully washed red phosphorus was used to convert the bromine thus obtained, after it had been several times re-distilled, into hydrobromic acid, and the hydrobromic acid was freed from iodine and organic matter by several fractional distillations with bromine water. From this pure hydrobromic acid, bromine was obtained by means of manganese dioxide free from chlorine. 2.10289 grms. (in vacuum) of silver yielded 3.66066 grms. (in vacuum) of argentic bromide on combination with this bromine,—a ratio of 57.445 : 100.00. Mr. Baxter found 57.444 in a similar experiment, while Stas's value was 57.445: hence the purity of our bromine and silver was proved.

Sodic hydroxide was freed from most metallic impurities (iron, &c.) by electrolysis. Ammonia was re-distilled in platinum vessels, as were also nitric and hydrochloric acids. Sulphuric acid was distilled in glass, alkalis being a less dangerous impurity than platinum in the instances where it was used. Water was purified by distillation,

first from alkaline permanganate solution, and then with a trace of acid potassic sulphate.

In processes where the presence of bromine rendered the use of platinum impossible, Jena glass, or at high temperatures Berlin porcelain, was used. For some of the platinum and other apparatus we are indebted to the Cyrus M. Warren Fund for chemical research in Harvard University.

The Method of Analysis.

Turning now to the method of analysing the carefully prepared nickelous bromide, it is obvious that the first point to be considered is the accurate determination of the weight of the salt to be analysed. This process was effected by means of apparatus similar to that devised for the drying and weighing of magnesic chloride, and described in a recent paper by Mr. H. G. Parker and one of us (*Proc. Amer. Acad.*, xxxii., 58), upon the atomic weight of magnesium. In this apparatus, constructed wholly of glass by Mr. Baxter, the bromide under consideration, contained in a platinum boat, was ignited at about 400° in a stream of mixed nitrogen and hydrobromic acid until constant in weight. It was then allowed to cool in a stream of pure dry nitrogen, and when cool it was pushed in pure dry air into its weighing bottle, which was immediately closed by a mechanical device. In this fashion it is possible to dry and weigh accurately the most hygroscopic of substances, and repeated ignitions of the same specimen have shown that perfect constancy in weight may thus be obtained. It is hard to believe that any water is retained by nickelous bromide at 400°, and certainly none could be absorbed during the cooling, for the whole apparatus was shut off from the outside air, and all the gases admitted were first passed through phosphoric oxide.*

The bromide in question was then weighed by substitution, using as the tare to be substituted a weighing bottle precisely like the one containing the platinum boat and substance. In this way alone can the weight of a large bottle be determined within the fraction of a tenth of a mgrm. The balance has already been described in detail (*Proc. Amer. Acad.*, xxvi., 242); the weights were of course compared and standardised with great care, and were used for no other work during the progress of this.

Having been weighed with accuracy, the nickelous bromide was dissolved in pure warm water in a flask, and from this was transferred to the large beaker flask in which the precipitation was to take place. The platinum boat in which the salt had been treated remained invariable in weight, showing that it had not been attacked by hydrobromic acid at a high temperature.

As has been said already, the salt used in the preliminary series was contaminated with a small amount of nickelous oxide, which was filtered off and weighed. The amount of this impurity is given simply to show that the slight irregularity of the results was not dependent upon the adulteration; the weights of nickelous bromide given are those left after the subtraction of the weight of the oxide. All the bromine contained in the solution was precipitated in these seven analyses by means of an excess of argentic nitrate, and the argentic bromide was collected and weighed with the usual precautions.

(To be continued).

Estimation of Small Quantities of Methyl-alcohol, Formic Aldehyd, and Formic Acid.—M. Nicloux.—The author has extended his method for the estimation of alcohol by the reduction of bichromate of potash in the presence of sulphuric acid, to the estimation of methyle alcohol, formic aldehyd, and formic acid.—*Bull. Soc. Chim. de Paris*, Series 3, xvii.-xviii., No. 16-17.

* A detailed description of this apparatus will be given in a future paper, upon Cobalt.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, November 18th, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

MESSRS. W. J. Elliott, H. S. Elworthy, F. F. de Morgan, Frank Moul, A. Harden, and Charles E. Brown were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Cecil Joslin Brooks, 24, Wood Street, Woolwich; Charles Henry Burge, Idesleigh Crescent Road, Kingston Hill, S.W.; M. J. Cannon, 101, The Chase, Clapham Common, S.W.; William Ransom Cooper, M.A., B.Sc., 87, Upper Tulse Hill, S.W.; Frederick Robertson Dodd, 1, Wesley Street, Liverpool; Jules Fuerst, 23, Marlborough Road, N.W.; James Brown Reid, 6, Southfield Terrace, Skepton; Harold Charles Sayer, Devon Villa, Summerhill Road, Dartford.

The PRESIDENT announced that he had received a letter from Sir Fleetwood Edwards stating that he had been commanded by the Queen to forward to him a Medal in commemoration of the 60th Anniversary of Her Majesty's reign.

Of the following papers those marked * were read :—

*118. "*On the Decomposition of Camphoric Acid by Fusion with Potash or Soda.*" By ARTHUR W. CROSSLEY and W. H. PERKIN, jun.

The authors find that by the action of fused potash on camphoric acid a very complicated mixture of acids is obtained. The volatile portion consists of acetic, propionic, isobutyric, isovaleric, and methylisopropylacetic acids, together with acids of the formulæ $C_6H_{13}CO_2H$, $C_7H_{15}CO_2H$, and $C_8H_{17}CO_2H$, of which the constitution is doubtful. The non-volatile acids consist of pimelic (isopropylsuccinic) acid and a new substance, dihydrocamphoric acid.

Dihydrocamphoric acid, $C_{10}H_{18}O_4$, crystallises in nodular masses melting at 105–106°, and on oxidation with dilute nitric acid yields succinic acid, oxalic acid, and an acid of the formula $C_8H_{14}O_4$, which is in all probability the $\alpha\beta$ -trimethylglutaric acid described by Balbiano.

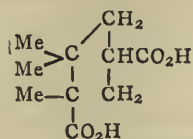
On treatment with acetic anhydride, dihydrocamphoric acid is converted into its anhydride, which, when heated to boiling, decomposes with elimination of carbon dioxide and formation of a cyclic ketone of the formula $C_9H_{16}O$. *Dihydrocamphoketone* is a liquid boiling at 180–181° and smelling strongly of peppermint. It forms a liquid ketoxime and a semicarbazone melting at 202–203°.

The results obtained on fusing camphoric acid with caustic soda differ markedly from the above. The lower volatile fatty acids appear to be the same as those obtained from caustic potash, but there is also present an unsaturated acid of the formula $C_8H_{15}CO_2H$.

The acids not volatile in steam consist of large quantities of pimelic acid and some unchanged camphoric acid, together with two new acids, pseudocamphoric acid, $C_{10}H_{16}O_4$, and an acid of the formula $C_9H_{16}O_4$, boiling at 254–257° at 50 m.m.

Pseudocamphoric acid crystallises from water in colourless six-sided plates with bevelled edges, usually grouped together in the form of stars, and melts at 119–120°. It forms a crystalline anhydride melting at 52–53° and an anilic acid melting at 208°. A further difference from its isomeride *d*-camphoric acid is that when treated with sulphuric acid it does not evolve carbon monoxide forming a sulphonic acid.

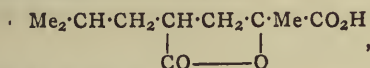
The authors explain the results of the action of fused alkalis on camphoric acid and deduce constitutional formulæ for the various compounds obtained on the assumption that camphoric acid is represented by the formula—



*119. "Experiments on the Synthesis of Camphoric Acid." By W. H. BENTLEY and W. H. PERKIN, jun.

The authors have attempted to prepare an acid of the constitution suggested (*Proc.*, 1896, xii., 189) as a probable formula for camphoric acid (see preceding abstract). They prepared isobutylmethylhydroxyglutaric acid,—

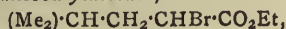
$\text{Me}_2\text{CH}\cdot\text{CH}_2\cdot\text{CH}(\text{CO}_2\text{H})\cdot\text{CH}_2\cdot\text{C}(\text{CO}_2\text{H})\text{Me}\cdot\text{OH}$, but did not succeed in eliminating water in the direction desired (at the points *), the product in most of the experiments being the lactone-acid—



or one of its derivatives.

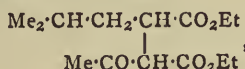
The following substances were prepared during the course of the work.

Ethylic bromisobutylacetate,—

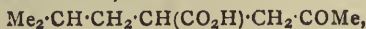


an oil boiling at 100—103° (17 m.m.).

Ethylic acetylisobutylsuccinate,—

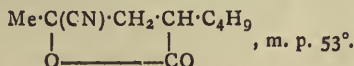


prepared by the action of ethylic bromisobutylacetate on ethylic sodioacetoacetate. It is a colourless oil boiling at 160° (25 m.m.), and when hydrolysed with dilute hydrochloric acid or sulphuric acid yields isobutylsuccinic acid, $\text{Me}_2\text{CH}\cdot\text{CH}_2\cdot\text{CH}(\text{CO}_2\text{H})\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$, m. p. 109°. When hydrolysed with concentrated hydrochloric acid, however, isobutyllevulinic acid,—

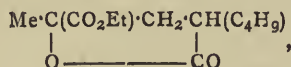


is formed. This is a colourless oil boiling at 190° (30 m.m.), and yielding a semi-carbazone melting at 192° bromine, in the presence of potash, oxidises it to isobutylsuccinic acid.

Isobutyllevulinic acid readily unites with hydrogen cyanide, forming isobutylhydroxycyanovaleric acid, $\text{Me}\cdot\text{C}(\text{OH})(\text{CN})\cdot\text{CH}_2\cdot\text{CH}(\text{C}_4\text{H}_9)\cdot\text{CO}_2\text{H}$, which crystallises with H_2O in needles melting at 95—96°. This acid on distillation yields the corresponding lactone,—



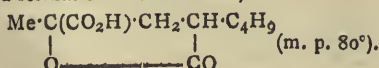
When an alcoholic solution of the hydroxy-cyano acid is saturated with hydrogen chloride, it is hydrolysed and converted into the ethereal salt of isobutylmethylhydroxyglutaric acid, $\text{Me}\cdot\text{C}(\text{OH})(\text{CO}_2\text{Et})\cdot\text{CH}_2\cdot\text{CH}(\text{C}_4\text{H}_9)\cdot\text{CO}_2\text{Et}$. On distillation, this loses alcohol, forming the ethereal salt of the lactone of isobutylmethylhydroxyglutaric acid,—



which is an oil boiling at 168° (17 m.m.). This on hydrolysis with alcoholic potash is converted into isobutylmethylhydroxyglutaric acid,—

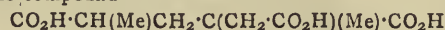


a crystalline acid melting at 134° with elimination of water and formation of the lactone,—

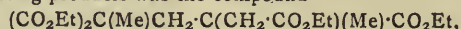


*120. "Synthesis of an Isomeride of Camphoronic Acid." By S. B. SCHRYVER, Ph.D.

The compound—



was synthesised by acting on methylacrylic ester, $\text{CH}_2\text{:C}(\text{Me})\text{CO}_2\text{Et}$, with sodiummethylmalonic ester. The addition product thus obtained, having the formula $(\text{CO}_2\text{Et})_2\text{C}(\text{Me})\cdot\text{CH}_2\cdot\text{CNa}(\text{Me})\text{CO}_2\text{Et}$, instead of being isolated, was acted on by iodacetic ester. Among the resulting products was the compound—

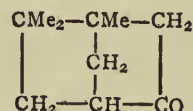
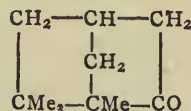


which on hydrolysis gave an acid of the required formula. This was isolated by means of its lead salt, which is insoluble in acetic acid, in the form of a syrup, and this on treatment with nitric acid gave a crystalline oxy-acid having the formula $\text{C}_9\text{H}_{14}\text{O}_7$. It is, therefore, an isomeride of camphoronic acid.

DISCUSSION.

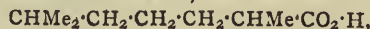
Dr. KIPPING said that it was very difficult to criticise papers which, like those read by Dr. Crossley, contained so many new and important facts relating to substances of somewhat complex constitution. It seemed to him, however, that all the results obtained by Dr. Crossley and Dr. Perkin in their study of the substances produced by fusing camphoric acid with potash, could be explained on the basis of Bredt's formula; such an opinion might of course be altered after carefully examining the details of the work as laid out in the published papers.

Dr. FORSTER pointed out that the formula for camphoric acid employed by Drs. Crossley and Perkin appears to involve the expression of the constitution of camphor by one of the formulæ—



The behaviour of camphoroxime on dehydration, and the isomerism of two series of campholenic derivatives, seem to render the second expression the more probable of the two, but before either could be accepted, an explanation must be furnished of the changes involved in the production of such compounds as cymene and carvacrol from camphor—changes which, it must be remembered, are readily explained by Bredt's formula.

The production of the acid,—



obtained by the authors by fusing camphoric acid with alkali, harmonises with the new camphoric acid formula, but it must not be overlooked that Bredt's formula for camphoric acid affords an equally plausible explanation.

Dr. CROSSLEY, in reply, said that the main evidence for accepting the formula proposed by Professor Perkin was embodied in his paper on sulphocamphylic acid, which had not yet been published in detail. Whilst the production of most of the compounds described by Professor Perkin and himself admit of explanation by Bredt's formula, it does not account for the formation of $\alpha\alpha\beta\beta$ -tetramethyladipic (dihydrocamphoric) acid.

*121. "The Action of Magnesium on Cupric Sulphate Solution." By FRANK CLOWES, D.Sc., and R. M. CAVEN, B.Sc.

The authors have examined the action of magnesium on solutions of cupric sulphate of different strengths, both at atmospheric temperature and at a temperature near their boiling-point. They find that the evolution of hydrogen which always takes place is accompanied by the precipitation of a mixture of cuprous oxide and metallic copper in proportions which vary with the conditions of the experiment.

When a dilute solution of cupric sulphate is employed, the above-mentioned products are accompanied by a

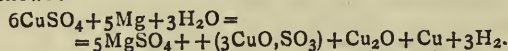
quantity of a green substance, which consists of a mixture of basic hydrated sulphates of copper and magnesium. This substance was observed to form when a saturated solution of cupric sulphate was employed, but it was decomposed again before the reaction was completed.

The time of the reaction varies from ten minutes in the case of a hot strong solution of cupric sulphate to several days, or even a week, when a dilute solution is employed at atmospheric temperature.

The quantities of the three reduction products, cuprous oxide, copper, and hydrogen, were determined under various conditions. A volumetric process depending upon the use of potassium permanganate was employed for the estimation of the cuprous compound when it occurred together with the basic sulphate of copper and magnesium before mentioned.

The authors found in each case that they investigated that the sum of the magnesium equivalents of the cuprous oxide, copper, and hydrogen obtained agrees very closely with the amount of magnesium employed in the experiment. The magnesium is therefore proved to have displaced from the solution of cupric sulphate, substances which are chemically equivalent to it, though only a small and variable proportion of these substances consists of metallic copper. The authors have shown that the nature of the reaction is not influenced by the presence of slight impurities in the cupric sulphate employed by carrying out similar experiments with a specimen of the salt obtained by six successive re-crystallisations of a sample which was originally almost pure. Pickering's observation of the formation of a basic sulphate of copper by the decomposition of a solution of cupric sulphate by boiling, has been incidentally confirmed, but the formula which the authors attribute to this compound is $4\text{CuSO}_4 \cdot 7\text{Cu}(\text{OH})_2 \cdot \text{H}_2\text{O}$.

NOTE—24th November.—The authors have now referred to the paper by Commaille (*C. R.*, 1866, lxiii., 556), and they find that an equation is given which is supposed to represent the reaction between magnesium and cupric sulphate solution. This equation, when it is put into modern form and corrected for an obvious error, reads as follows:—



No details are given as to the temperature or the strength of the cupric solution which acted on the magnesium, nor are the analytical data stated upon which the above equation is founded. It will be noticed that the formation of basic magnesium compounds is not accounted for in the above equation. The authors consider that the results of their own analyses and examination prove that the composition of the basic sulphate of copper is variable, but that it does not correspond to the above formula, and certainly occurs in the condition of hydroxysulphate. They have never found that the proportion between the quantities of cuprous oxide and metallic copper is such as can be definitely represented by means of an equation, but rather that it varies within wide limits under different experimental conditions.

It will therefore appear that Commaille's imperfect study and statement of the reaction may well be supplemented by a more complete investigation.

The authors desire to place on record the fact that, when zinc acts upon a cold solution of copper sulphate, small bubbles of gas are evolved; and that when a hot solution of cupric salt is employed, an appreciable quantity of hydrogen can be collected, and cuprous oxide is found in the residue. They intend to study this reaction in detail.

DISCUSSION.

Professor TILDEN remarked that the work of the authors had been anticipated by the experiments of Commaille, who had published thirty years ago the results of an enquiry into the action of magnesium on neutral metallic salts, including copper sulphate.

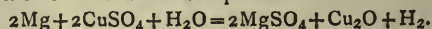
When magnesium is immersed in an aqueous solution of copper sulphate free from acid, the action seemed to take the following course:—First, there was a precipitation of spongy metallic copper, which, in contact with the magnesium, gave a couple capable of decomposing water at common temperatures. Hydrogen was then evolved, and a crust of magnesia formed on the surface of the metal. The copper salt was locally reduced by the hydrogen to the cuprous state, and this, in the presence of the magnesium oxide, led to the precipitation of cuprous oxide and of a basic cupric salt. The green precipitate formed therefore consists of magnesia and basic cupric salt, the proportion varying according to the temperature and strength of the solution. This hypothesis is borne out completely by the results both by Commaille and the authors of the paper, and serves to account for the apparent inactivity of the magnesium which is mechanically protected by the crust which forms upon its surface. The speaker had tested the suggestion that the metal was rendered inactive by a film of hydrogen and had come to the conclusion that this was not the case.

Professor CLOWES said that the origin of the investigation dated back some two years; the irregular action of magnesium on the cupric solution having been noticed in the course of experiments made in connection with a laboratory curriculum which he was then drafting for elementary students. It was anticipated that the reaction of magnesium on cupric sulphate might serve to establish the relative chemical values of copper and magnesium, but this was found to be impracticable owing to the evolution of a large amount of hydrogen. If such irregular actions occurred, instead of the simple replacement of one metal by another in solutions of its salts, it would seem that this method of determining chemical equivalents was not of general application.

Mr. CAVEN, in reply, said he regretted that he had overlooked the paper by Commaille on this subject to which Dr. Tilden referred.

It seems improbable that cuprous salt is formed by the reducing action of nascent hydrogen upon the cupric salt in the solution. This would lead to the formation of free sulphuric acid, which would decompose the cuprous oxide formed at the commencement of the reaction into cupric sulphate and metallic copper. If nascent hydrogen reduces the cupric salt at all, it can only produce metallic copper. Sulphuric acid would be liberated in this case also, though not in immediate contact with cuprous oxide. It may be that the process of solution of the magnesium hydroxide and basic copper salt which occurs at the close of the reaction in concentrated solutions, depends upon the action of the free acid thus produced.

The authors suggest that the magnesium reduces the cupric solution directly, producing cuprous oxide and hydrogen in the following manner, the proportion of the hydrogen evolved in the gaseous state being dependent upon the conditions of the experiment:—



Some such reaction as this, proceeding simultaneously with the action of the couple, may account for the immediate formation of cuprous oxide, and the vigorous evolution of hydrogen at the moment of immersion of the magnesium. The action of the magnesium-copper couple on the water is not in itself sufficient to account for the very large quantity of hydrogen which is obtained, or for the rapidity with which it is produced, since, when the couple acts upon pure water, the hydrogen is evolved only very slowly. This view of the origin of the cuprous oxide is borne out by the fact that it occurs, together with metallic copper, quite from the commencement of the reaction, and that the formation of hydrogen appears to begin directly the magnesium is plunged into the solution. This would not be the case if the formation of a metallic couple were necessary for the evolution of the gas.

There is no cessation in the evolution of gas during the course of the reaction under any circumstances. The

length of time necessary for the completion of the reaction in dilute solutions is undoubtedly due to the mechanical protection of the magnesium by the deposit, as Professor Tilden suggested.

*122. "Properties and Relationships of Dihydroxytartaric Acid." By HENRY J. HORSTMAN FENTON, M.A.

Bearing in mind the highly interesting constitution of hydroxytartaric acid, and the close relationship which has been shown to exist between this acid and dihydroxymaleic acid, a further investigation of its properties appeared to be desirable, especially as the free acid appears to have been scarcely studied.

It is now found that the free acid may very easily be prepared in a pure state by oxidation of dihydroxymaleic acid in presence of water. The yield is over 70 per cent of that demanded by theory. The dry acid shows no tendency to lose water at 90°. In aqueous solution, the acid decomposes when heated into tartronic acid and carbon dioxide. This reaction affords a very convenient method for the preparation of pure tartronic acid, the yield being 97 per cent of that theoretically obtainable. On titration with alkalis at 0° dihydroxytartaric acid behaves normally as a dibasic acid. But the results obtained at the ordinary temperature with caustic alkalis, using phenolphthalein as indicator, are considerably higher, being intermediate between those required for a di- and a tri-basic acid. These high results are shown to be due to the partial decomposition of the salts formed into tartronates and carbon dioxide.

By careful treatment with certain reducing agents dihydroxytartaric acid may be reduced to dihydroxymaleic acid or its isomeride. Zinc, in calculated quantity, and dilute acid reduces it to the β -form. Hydrogen bromide reduces it to dihydroxymaleic acid (α -form) with liberation of free bromine.

The reaction $C_4H_4O_6 + 2H_2O + Br_2 = C_4H_6O_8 + 2HBr$ is in fact a reversible one, the final distribution depending upon the masses of the reacting substances, and perhaps somewhat on the temperature.

*123. "The Molecular Association of Liquids and its Influence on the Osmotic Pressure." By HOLLAND CROMPTON.

In a paper which I recently had the honour of bringing before this Society (*Trans.*, 1897, lxxi, 925), I contended that the molecular association of liquids exercises an influence on their osmotic pressure, and endeavoured to show how this influence may be taken into account. My attention has been drawn to the fact that Planck, more especially, has long since proved that association could have no effect on the osmotic pressure of liquids (see the discussion between Planck and Wiedemann, *Zeitsch. Physik. Chem.*, 1888, ii., pp. 241, 343). This being the case, I wish to offer the following brief criticism of Planck's results.

In his "Vorlesungen über Thermodynamik" (Leipzig, 1897), Planck deduces, on p. 235, the following formula for the osmotic pressure of a dilute solution:—

$$P = \frac{R\theta}{n_0 m_0 v} (n_1 + n_2 + n_3 + \dots).$$

Here R is the constant of the gas equation, θ the absolute temperature, $n_1 + n_2 + n_3 + \dots = n$ the number of dissolved molecules, n_0 the number of molecules of the solvent, m_0 its molecular weight, and v its specific volume (volume of unit weight). Since $n_0 m_0 v = V$ is practically the whole volume of the solution $P = R\theta n/V$. This is, of course, van't Hoff's equation for the osmotic pressure, and it is argued that the product $n_0 m_0 v$ being simply the total volume of the solution, the pressure is independent of any change produced by association in m_0 . In other words, as long as V is constant P will remain constant, no matter what the value of m_0 .

The nature and derivation of this relationship will perhaps be best understood if we suppose that we have two gases, and that n molecules of the first are dissolved in,

or mixed with, n_0 molecules of the second. The partial pressure of the first gas in the mixture is $n/(n_0 + n)$ of the total pressure, or if n is small in comparison with n_0 , we may say n/n_0 of the pressure of the second gas. The pressure of this latter is RT/V_0 , and, in order that R may have a fixed value, let V_0 be the molecular volume $m_0 v_0$ of the gas, where m_0 is its molecular weight, and v_0 its specific volume. The partial pressure of the dissolved gas then becomes $p = RTn/n_0 m_0 v_0$, or, since $n_0 m_0 v_0 = V_0$, whole volume of the solvent gas, $p = RT/nV_0$.

These equations for the partial pressure of a gas present in a dilute state in any gaseous mixture are identical in form with those of Planck for the osmotic pressure of dilute solutions. It follows, then, that we have apparently only to liquefy the solvent gas, to substitute the molecular volume of the resulting liquid solvent for that of the gaseous solvent, and the partial pressure of the dissolved gas then becomes its osmotic pressure in the solution.

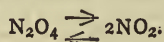
The result is not surprising, for it is obtained by a practical reversal of Planck's line of argument. It will be found (*loc. cit.*, p. 215) that, in his treatment of the thermodynamics of dilute solutions, he adopts the view that any such solution could be completely gasified without any change in the values of n and n_0 , and, of course, the osmotic pressure of the dissolved compound in the solution would then become the partial pressure of the dissolved gas in the gaseous mixture. But the view that a solution could be completely gasified without any change of a permanent character in the values of n and n_0 cannot be regarded as justifiable in the light of all the recent work on the molecular condition of liquids. And although it is not surprising to find that, starting with this view, Planck subsequently comes to the conclusion that association of the solvent does not influence the osmotic pressure of a solution, the reasoning evidently moves too completely in a circle to carry much conviction with it.

For, in order to ascertain what effect a change due to molecular association in the solvent would have on our formulæ, let us first take the case of the mixed gases, and suppose the gas we term the solvent to be one, say, like nitrogen dioxide, NO_2 , which undergoes association as the temperature falls. Such association will bring about a change of m_0 to xm_0 , of n_0 to n_0/x , and of v_0 to v_0/x , if x represents the degree of association (factor of association) for any temperature T. The partial pressure of the dissolved gas now becomes $p = xRTn/n_0 xm_0 v_0$, or since $n_0 xm_0 v_0 = V_0$, $p = xRTn/V_0$. Hence, although when a gas is dissolved in any other gas, and is present only in a dilute condition, its partial pressure will, under normal conditions, follow the law that $pV_0 = nRT$ if association of the solvent sets in, the law for perfect gases no longer holds, but the relationship becomes $pV_0 = xnRT$ where x represents the factor of association of the solvent for the temperature T.

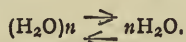
In fact, the partial pressure of the dissolved gas is, under the above conditions, dependent on the volume occupied by the solvent. Under normal conditions the solvent obeys the usual gas equation, and the partial pressure therefore alters in accordance with this relationship. But if association or dissociation of the solvent sets in, the gas equation will not immediately apply, but will require a suitable modification, and this modification will hold also for the partial pressure formula.

Now the osmotic pressure of a dilute solution presents an analogous case. As long as the solvent is a monomolecular one we may accept Planck's reasoning, and hold that the osmotic pressure follows the law $P = nR\theta/n_0 m_0 v$. But suppose that the solvent begins to undergo association, so that for any given temperature θ a change of m_0 to xm_0 has taken place. At the same n_0 changes to n_0/x , and a simultaneous change will be found to have taken place in v . This last change has, it seems to me, been entirely overlooked up to the present, although any one who gives the matter a moment's consideration cannot fail to admit that an associated liquid would not have the density of the same compound in the

monomolecular condition. All the evidence at present points to the conclusion that association increases the density of a liquid, and that consequently association would alter the value of v to v/a . The precise magnitude of a cannot at present be ascertained (unless perhaps with the aid of Traube's investigation of the molecular volume), but its value is probably not far removed from that of x . The osmotic pressure, then, of any dilute solution, the solvent of which is undergoing association, would be given by $P = anR\theta/n_0m_0v$, or, since $n_0m_0v = V$, by $P = anR\theta/V$, an expression that is similar to the partial pressure formula. While, then, the osmotic pressure of any substance in solution in a monomolecular solvent follows the gas equation $PV = R\theta$, if the solvent is one which is undergoing association or dissociation, the equation for the osmotic pressure becomes $PV = aR\theta$. And here another point which has apparently always been overlooked by those who contend that association of the solvent does not influence the osmotic pressure may be alluded to. It has been usual, hitherto, to compare with one another two stable systems, one of which is assumed to contain a monomolecular solvent, and the other the same solvent in the associated condition, and to show that the gas equation $PV = R\theta$ applies to each. The comparison, however, should not be between a monomolecular and an associated solvent, but between a monomolecular and an associating solvent, *i. e.*, one in which the association is continually altering with the temperature. The gas equation would hold for the gas NO_2 , and it would hold equally for the gas N_2O_4 , but it will not apply throughout the range of temperature in which—



The gas equation might in like manner hold for the osmotic pressure of a substance in solution in water of the molecular weight 18, and it might also hold for water of the molecular weight 3×18 , but water, as we know it, is neither of these compounds. It is a substance the association of which is continually changing with the temperature, and the attempt is therefore being made to apply the gas equation in the range of temperature throughout which—



Van Laar has, like Planck, treated the osmotic pressure of dilute solutions from the thermodynamical standpoint, and confirmed the latter's conclusion that association of the solvent does not influence the osmotic pressure. I quote here literally what Van Laar says on this matter (*Zeit. Physikal. Chem.*, 1895, xviii., 274):—

"We found that the osmotic pressure $\pi = R\sigma(x+a)/va'$, where $\sigma(1+a)$ has the value Σc ,* and va' , the volume of 1 grm. molecule of the pure solvent. For water va' was therefore given the value 18. If, however, association occurs, the weight of the grm. molecule would be $18a$, so that for va' the volume of $18a$ grms. of water must be substituted. Calling this last va' , then $Va' = av_a'$. But also σ , the concentration of the dissolved substance, becomes a times greater. For we have now no longer one molecule of the dissolved substance to n molecules of water (taking the mol. wt. of water = 18), but one molecule to n_1/a molecules of water, so that the concentration must be expressed by an a times greater number than before. Calling this C , then as $C = a\sigma$, the expression—

$$\pi = R\sigma C(1+a)/Va'$$

does not differ from $\pi = R\sigma(x+a)/va'$, where the quantities va' and σ are independent of the association."

The italics in the above are mine, for it is this portion of the paragraph that I wish to direct attention more especially. The molecular volume of any compound is given by M/d , where M is the molecular weight of the compound and d its density. Now Van Laar supposes

that if liquid water had the molecular weight 18, its molecular volume would be $18/1$; or if it had the molecular weight $18a$, its molecular volume would be $18a/1$. In other words, the same density is to be assigned indiscriminately either to the monomolecular or to the associated compound. But can any one doubt if liquid water were obtainable having the molecular weight 18, that it would most certainly not have the density 1 at ordinary temperature? Take water merely as the first term of the $\text{C}_n\text{H}_{2n+1}\text{OH}$ series of alcohols, a series the members of which are themselves associated, but not to the same extent as water, and analogy at once points to a much lower density than 1 for monomolecular liquid water, a density that is in fact probably not far removed from $1/a$. In fact there is absolutely no evidence that the molecular volume of an associated liquid does differ greatly from that of the same liquid compound in the monomolecular state. Van Laar's argument *against* is converted therefore into an argument *for* the effect of association on the osmotic pressure.

PHYSICAL SOCIETY.

Ordinary Meeting, December 10th, 1897.

Mr. SHELFORD BIDWELL, President, in the Chair.

Mr. ALBERT CAMPBELL exhibited (1) An experiment to illustrate alternate exchange of Kinetic Energy. Two brass spheres, each about 1 inch diameter, are suspended from the same point by equal wires. One of them is then thrown so as to describe a circular orbit. The second sphere, starting from rest, gradually takes up motion from the first sphere, and in turn describes a circular orbit. The first now comes to rest, and the reverse process takes place. This alternating action repeats itself until all the energy is lost in the wires. (2) An experiment to illustrate the Low Heat-conductivity of Glass and the Expansion of Glass by Heat. A long tube is clamped at the lower end, in a vertical position. One side of it is then heated with the flame of a Bunsen burner, and the glass is observed to bend, moving over a fixed mark near the top of the tube. When the flame is withdrawn, the first position is quickly regained.

Mr. CAMPBELL then read a paper on "Temperature Compensators for Standard Cells."

Some account of the methods adopted by the author has already been published; he now describes the apparatus. The first compensating arrangement (3) can be used for keeping the potential-difference between two points of a conducting system constant at all room-temperatures. Or it can be adapted to modify the voltage of a standard cell to some convenient whole number. This arrangement (3) resembles a Wheatstone's bridge with the galvanometer-branch removed. One pair of opposite arms is of copper, the other pair is of manganin. The bridge-battery is a Leclanché cell: this supplies the auxiliary voltage, which is utilised at the two galvanometer-points of the bridge, and is there applied in series with the standard cell. In an alternative method, suggested by Mr. C. Crawley, only one of the four arms is made of copper. The second compensating arrangement (4) is intended to maintain constant potential between two points, at all room-temperatures. For this purpose two wires, a and b , are connected in parallel. One of them, a , is all of manganin; the other, b , is partly copper and partly manganin. Constant current is applied at the ends of a and b . The various resistances are chosen so as to give constant difference of potential between the ends of the manganin portion of b . By this method the potential difference can be maintained to within 1 in 2000.

Mr. SWINBURNE said that twelve or thirteen years ago he had given a good deal of thought to compensation by wires of different temperature-coefficients. The first thing he tried was a Wheatstone's bridge. This was

* The c here is the n of the Planck formula.

compensated by making the bridge-arms of wires whose temperature-coefficients differed—as, for instance, platinum and copper. He then applied the same principle to the compensation of standard cells, using a potentiometer method that gave direct readings, and to the compensation of voltmeters and Watt-meters. These results were published between 1885 and 1890, in the electrical journals. He believed that Mr. Evershed had also developed this idea, by putting "back" turns on voltmeters, and by other differential devices. The details of Mr. Campbell's apparatus had a few points of special interest. The way in which he connected up the bridge (3) seemed particularly worthy of notice.

Prof. AYRTON asked whether thermo-electric effects produced difficulty in the compounded arrangement.

Mr. CAMPBELL said the system was symmetrical, and the thermal currents were consequently neutralised.

Mr. APLEYARD, referring to experiment (2), said it was identical with one that had been shown for the past eight years at lectures at Cooper's Hill College. It was specially interesting as illustrating the deflection that occurs with girders and bridges when exposed on one side to sunshine.

Mr. J. ROSE-INNES read a mathematical paper on "Lord Kelvin's Absolute Method of Graduating a Thermometer."

Lord Kelvin has investigated the cooling effects exhibited by various gases in passing through a porous plug. He found that for any gas, kept at the same initial temperature, the cooling effects were proportional to the difference of pressure on the two sides of the plug. He also found that, for any one gas, the cooling effect per unit difference of pressure varies approximately as the inverse square of the absolute temperature. This rule holds very well in the case of air; it is not so satisfactory for carbonic acid; it fails for hydrogen. With hydrogen there is a heating effect that increases, if anything, when the temperature rises. Mr. Rose-Innes proposes an empirical formula, containing two disposable constants, α and β , characteristic of the gas in question. Denoting by T the absolute temperature, he finds that, very approximately, the cooling effect is given by the expression—

$$\left(\frac{\alpha}{T} - \beta\right).$$

This relation includes the three cases—air, hydrogen, carbonic acid—under one form, and thus enables them to be treated in one common investigation. Moreover, the differential equation concerned in the thermo-dynamic scale is thereby rendered more manageable,—it leads to simple algebraic results after integration. The paper discusses the thermo-dynamic correction for a constant-pressure gas-thermometer, and the correction for a constant-volume gas-thermometer; also an estimate of the absolute value of the freezing-point of water: the results obtained take, for the most part, a very simple shape, using the above expression for the cooling.

Dr. S. P. THOMPSON said the empirical expression,—

$$\left(\frac{\alpha}{T} - \beta\right),$$

indicated that at some particular temperature the cooling effect vanished; that was a point suggestive of useful results if investigated by experiment.

Mr. J. WALKER read a communication from Mr. BAYNES on the paper, and remarked on the desirability of adopting two constants. He thought that further experiments should be made to discover how specific heat at constant temperature depends on temperature. The calculated values for hydrogen were too few to be taken as evidence of the validity of the rule.

Mr. ROSE-INNES, in reply, said that from what was known of hydrogen, it might be expected to behave at ordinary temperatures as air behaves at higher temperatures. His object was, if possible, to include in one

formula the case of the three investigated gases. This was much better than having a separate formula for each gas. Whether or not hydrogen was conformatory with air and carbonic acid might be considered as *sub judice*; it required further experimental data to test the formula in that case.

The PRESIDENT proposed a vote of thanks to the authors, and the meeting was adjourned until January 21st, 1898.

SOCIÉTÉ D'ENCOURAGEMENT POUR L'INDUSTRIE NATIONALE.

November 26, 1897.

M. MASCART, President, in the chair.

The SECRETARIES read several letters received, after which M. GROIGNARD exhibited an automatic valve for the arrest of noxious fumes, and an automatic water level control-valve of similar construction.

M. A. POUCHET exhibited an apparatus for aerial navigation.

Several gentlemen were proposed as candidates for the position of member of the committee on Mechanics, and two candidates were announced as having been elected members of the Society.

An interesting paper was then read by M. BECHMANN on the "Purification of the Seine," for which he received the thanks of the Society. This paper will be printed in the *Bulletin*.

The next meeting for the election of officers for 1898 was announced to take place on December 10, 1897.

NOTICES OF BOOKS.

A Complete Catalogue of Practical Physical Apparatus Manufactured by W. J. GEORGE, late BECKER & Co.

THIS catalogue contains a complete list of the Physical apparatus recommended by Professor Schuster and Dr. Lees in their text-book on "Practical Physics," and will doubtless be found to be of great assistance to both teachers and students not already provided with the necessary apparatus for their studies. It is a great advantage and saving of time to be able to order any particular piece of apparatus which may be required, with a knowledge that it is kept in stock and can be delivered at short notice, thus avoiding those tiresome delays to which we are too much accustomed. The list appears to be comprehensive, well illustrated, and conveniently arranged.

OBITUARY.

DR. CAMPBELL MORFIT.

By the death of Dr. Campbell Morfit, M.D., formerly Fellow of the Chemical Society and of the Institute of Chemistry, which took place on the 8th inst. at South Hampstead, the science of applied chemistry has suffered a severe loss. An American by birth, he had for many years past been a London resident. In 1854 he was appointed Professor of Applied Chemistry in the University of Maryland, and was one of the scientific advisers of the United States Government previous to the Civil War.

He was the author of several standard works on industrial chemistry, including "Chemical and Pharmaceutical

Manipulation," "Arts of Tanning and Currying," "Oleic Soaps," and with Dr. James C. Booth was joint editor of the "American Encyclopedia of Chemistry."

His work included researches on the the guanos, salts, sugars, analyses of coals, gum mesquite, and glycerin. During the latter period of his life his attention was devoted to the improvement of technical processes, the preparation of condensed food rations, the manufacture of paper, and in especial to the questions of the refining of cotton and linseed oils and the utilisation of cotton-seed oil as a food substance.

His loss will be deeply felt by a wide circle of scientific and literary friends.

CORRESPONDENCE.

SEPARATION OF NICKEL AND COBALT FROM IRON.

To the Editor of the Chemical News.

SIR,—May I take exception to the opening statements of the article on the separation of Ni and Co from Fe, &c., which you reprint in the CHEMICAL NEWS (vol lxxvi., p. 279).

To say that there is no satisfactory method known for effecting the separation of Ni and Co from large quantities of Fe is surely untrue.

The following, if test analyses are to be credited, are a few references to satisfactory methods already described in your journal:—

1. Precipitation of the iron with lead oxide (Field, CHEM. NEWS, i., 4).
- 2 and 3. Precipitation as ferric phosphate in acetic solutions (Moore, CHEM. NEWS, liv., 300; Campbell, lix., 139).
4. Treatment with excess of soda pyrophosphate; whereby, even in presence of much iron, a clear colourless solution is obtained, which is subsequently titrated with cyanide of potassium (Moore, CHEM. NEWS, lix., 292).
5. Conversion of the iron into ferricyanide, Ni into nickelocyanide, and subsequent precipitation of Ni by a solution of bromine in potash (Moore, CHEM. NEWS, lvi., 3).

This is not a complete list of the processes to be found in the CHEMICAL NEWS. Other well-known and largely used processes are to be found in text-books specially devoted to steel analysis. Two other processes which have been well tested and find great favour are Campbell and Andrew's xanthate process (*Journ. Amer. Chem. Soc.*, Feb., 1895), and Herschell's hydrate process ("Fresenius's Quantitative Analysis," p. 437).

The second paragraph specifies hydrate and basic acetate precipitates as "always containing a considerable proportion of other metals (Ni, Co, Mn, Cu, &c.)."

The case for acetate precipitations has already been presented at sufficient length. It is necessary only to refer to the separate papers—Nickel, p. 49; Cobalt and Manganese, p. 165; Copper, p. 210—to see that respecting acetate the charge is unjustly made.

It is not claimed that any of the above processes are more accurate than the electrolytic process, although some of them, with their accompanying estimations, are equally so. But when the employment of the process in everyday practice comes to be considered, their superiority in point of quickness and general convenience is most marked.—I am, &c.,

HARRY BREARLEY.

THE DAVY FARADAY RESEARCH LABORATORY OF THE ROYAL INSTITUTION.

Directors:

The Right Hon. LORD RAYLEIGH, M.A., D.C.L., LL.D., F.R.S.,
Professor DEWAR, M.A., LL.D., F.R.S.

Superintendent of the Laboratory:
Dr. ALEXANDER SCOTT, M.A., D.Sc.

This Laboratory, which has been founded by Dr. Ludwig Mond, F.R.S., as a Memorial of Davy and Faraday for the purpose of promoting original research in Pure and Physical Chemistry, is now open.

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The above results must be taken only as approximation, for this reason:—Each sample and the standard were treated with equal amounts of cyanide, and therefore

"0.05" contains a larger excess than the standard, and "0.10" a still larger. This would (if the influence of the excess of cyanide on the copper is the only consideration) make the results somewhat high; "0.10" more so than "0.05." A corrected result might even show the proportional action of increasing amounts of chromium which is suggested by the given figures. Elsewhere this source of error has been guarded against.

I am unable at present to propose a probable explanation of the unexpected results; but I am able to point out that the chromium existing as chromic acid exerts no material influence, and thence an obvious means of overcoming the difficulty.

The yellow colour of the chromate does not very greatly interfere with the colour observation. The change is from green to a pure yellow.

Cobalt, Nickel, Silver.—These were not done. They would interfere in a regular way, and if estimated by other means could be allowed for in titrating a mixture. Thomson found it impracticable to estimate copper in presence of large quantities of cobalt. With only colour changes to rely on, this is undoubtedly the case. Field states the interference to be 12 per cent. This only serves to confirm Thomson's statement, because the interference of cobalt is really as great as that of nickel. An explanation of how this 12 per cent was arrived at would be interesting. Thomson finds nickel to interfere 120.4 per cent; Field, 95 per cent. This difference, and some others, might be accounted for by supposing that the amount of alkali and alkali salts varied with each operator if it were not for the striking agreement in their values for silver—31.7 and 32.0.

Gold, platinum, and palladium are omitted on account of their cost. Thomson finds them to interfere 32.65, 19.25, and 62.45 per cent respectively. Such large interferences suggest the possibility of estimating them by some modification of the cyanide titration.

Tin.—Precipitate with either alkali; not easy to filter. The effect of stannous salts was not quantitatively measured; they cause low results.

Molybdenum (Soda Molybdate).—No precipitate in either case.

Arsenic (Soda Arsenate).—No precipitate in either case.

Antimony.—A precipitate with either alkali; easily filterable.

Bismuth (Nitrate).—White precipitate; moderately filterable.

Lead (Acetate).—Precipitate with either alkali; moderately filterable.

Mercury (ous).—Final precipitate grey; only slight in ammoniacal solution, more with soda; easily filtered.

Mercury (ic).—No precipitate in either case after titration. It is noticeable that the influence of mercury is fairly regular. Denigés, who has based so many delicate analytical methods on the $\text{AgI} + \text{KCN}$ reaction, has recently (*Journ. Chem. Soc., Abs.*, 1897, ii., 433) applied it to the estimation of mercury.

Uranium (Nitrate).—Soda: No precipitate. Ammonia: Yellow precipitate, which does not dissolve on adding the cyanide, unless the cyanide contains considerable carbonate; filters rather badly.

Separating and Estimating Copper.

Precipitation of copper as subsulphocyanide by sulphurous acid and a sulphocyanide is a well-known means of separating copper from other elements. Hugo Tamm (*CHEMICAL NEWS*, xxiv., 91; Crookes, "Select Methods," p. 264) uses a mixture of equal weights of sulphocyanide and soda or ammonia bisulphite, and claims by these means to separate copper from whatever substance may accompany it. As the above table does not include all substances whatever, and as no means are suggested for avoiding some of the interferences, I gladly draw attention to Tamm's reagent. So far as I have had occasion to use it, or rather its like (H_2SO_3 and KCNS), the separations have been very satisfactory. If the estimation of the

copper were as easy as its separation, all would be well, but at the very next operation it behaves badly by passing through the filter. The subsequent drying of the filter is lengthy, and thus an admirable separation is largely overlooked on account of the difficulties in making the estimation.

The following recommendations are made to meet this difficulty:—

Having precipitated the copper, decant the solution on to a small asbestos or paper pulp filter; if necessary, the filter may be washed with a dilute solution of the precipitating reagent and then replaced in the precipitating flask. Add a few c.c. of nitric acid, 20 c.c. 2/N hydrochloric acid, and boil for a few minutes. The copper and any adhering sulphurous acid are oxidised, and the cyanogen compounds are decomposed. Cool, neutralise with soda carbonate, add the usual excess, and titrate as usual.

No test analyses are given. The analyses of nickel, copper, and zinc alloys, to be given in a subsequent paper, will serve this purpose equally well.

The Laboratory,
Norfolk Works, Sheffield.

THE EXACT WEIGHT OF OXYGEN, HYDROGEN, AND NITROGEN.

By the Author of "Reform of Chemical and Physical Calculations."

PROFESSOR ARMSTRONG said at the Chemical Society, March, 1896: "A pure substance is, and must ever remain, an ideal conception; we should not speak of a substance as pure, when such a condition of matter is known to be unattainable." If this be true (and many eminent chemists endorse that sentence), how can we rely upon experiments which give, for instance, the weight of hydrogen with five decimals when we know that the hydrogen we can prepare is *not pure*? even filtered through a diaphragm of hot platinum, it shows a "compound" spectrum (*CHEMICAL NEWS*, vol. lxxvi., p. 170), and as hydrogen is the lightest substance known, every admixture must increase its weight.

The weight of oxygen can be, and no doubt is, determined more exact than that of any other gas; and if Lord Rayleigh's determination of the weight of 1 litre oxygen = 1.42952 grm., is taken as correct for the latitude of London, the experimental determination of hydrogen, 1 litre = 0.09001 grm., is *more* than 1/16 of the weight of oxygen, and so are the results found by all other investigators; 1.42952/16 is only 0.089345 grm. The difference between the experimental and the calculated value is 0.090010 - 0.089345 = 0.000665 grm. per litre H; and such a difference would be caused if 0.39 c.c. argon are mixed with 999.61 c.c. pure hydrogen, or 0.2 c.c. argon + 0.3 c.c. N mixed with 999.5 c.c. pure hydrogen.

If pure N has the relative weight 14, O being = 16, then a mixture of 999 c.c. N + 1 c.c. argon would be = 14.005,—the value found by M. Leduc's latest experiments. If chemists can *prove* that they are able to detect and remove these or other impurities, if they further can *prove* that their instruments and observations are absolutely correct, and that every experimenter gets the *same* result, then they may dispute the relative weights $\text{H} = 1$, $\text{N} = 14$, $\text{O} = 16$.

C. J. T. HANSEN.

Copenhagen, December 11, 1897.

The Astronomer-Royal of the Berlin Observatory, who has closely studied my "Reform," has authorised me to publish the following:—

(TRANSLATION).

Berlin, Sternwarte.

... My opinion is, that such chemical and physical investigations as are developed in your "Reform of Calculations," quite apart from the ordinary course of research and study, not only now are of high importance, but ultimately may lead to very great and eminently practical simplifications of scientific work and study, and therefore are worthy of public acknowledgment and promotion.

(Signed),

Professor WM. FOERSTER, Dr. phil.

Note to Review, CHEMICAL NEWS, August 6th, 1897, page 70.—The "Carlsberg Foundation" is one of the principal institutions for the advancement of science in Copenhagen, and administered by Professors of the University. My "Reform" is printed under the patronage and at the expense of that Institution, not by the Carlsberg Foundation, as stated in the CHEMICAL NEWS.

ERRATA.—CHEMICAL NEWS, November 26, 1897, p. 264, col. 1, instead of "0.42952" read "1.42952"; and instead of "56/10" read "56/80."

QUALITATIVE RESEARCH ON TRACES OF ALKALINE CARBONATES IN PRESENCE OF AN EXCESS OF BICARBONATES, OR OF BORAX.

By ALEX. LEYS.

FOR the purpose of differentiating between the bicarbonates and the alkaline carbonates we make use of a salt of magnesium, such as the sulphate, for example: the neutral carbonates give a white precipitate, while with bicarbonates the solution remains clear.

This reaction, which gives excellent results when applied to either one or the other of these salts, does not answer when they are mixed. Such is the case with certain products which are met with commercially, under the name of "milk preservers." In them we find mixtures of carbonate and bicarbonate of sodium, of carbonate of sodium and borax, or all three of these salts together.

From several analyses we have made we have found that with a mixture of crystallised bicarbonate and carbonate of sodium, in the proportion of 32 of the former to 68 of the latter, completely dissolved in water, no precipitate was formed with sulphate of magnesium, from which it might be concluded, relying only on this reaction, that the substance was formed of bicarbonate of sodium only. In the same manner a mixture of crystallised carbonate of sodium, with a sufficiently high percentage of borax, such as 40 of borax to 60 of the neutral carbonate, does not precipitate sulphate of magnesium, and leads one to believe in the same way, without further information, that the substance under examination is nothing but bicarbonate.

It is thus seen that a small quantity of bicarbonate or of borax may hide a considerable proportion of neutral carbonate. We have therefore sought for a more trustworthy and sensitive reaction. A saturated solution of sulphate of lime seems to fulfil our requirements.

Such a solution remains limpid during a certain time only when we add to it pure bicarbonate of soda; then we see gradually appear, throughout its mass, microscopic crystals which eventually make the liquid cloudy. With pure borax we also do not observe any cloudiness when the solutions are first mixed, and, further, the liquid does not become cloudy on standing; but as soon as the smallest quantity of neutral carbonate is present with one or the other salts in solution, the former, when added to a solution of sulphate of lime, immediately gives rise to a

heavy, opaque, white precipitate of carbonate of lime. This reaction is so sensitive that if we pour over a mass of commercial bicarbonate of soda, usually sold as pure, a quantity of water insufficient to dissolve it entirely, we immediately obtain with this first solution, when added to sulphate of lime, the white heavy precipitate characteristic of the neutral carbonate. If we throw away the first water, and add fresh, the second solution will often give the immediate precipitate, and it is only at the third addition of water that the presence of the neutral carbonate no longer shows itself, and that we usually get a solution of pure bicarbonate of soda.

It is easily seen from this, that if we use both sulphate of magnesium and sulphate of calcium, we cannot, when dealing with a mixture, be mistaken as to the nature of its constituents.

It will suffice, when the sulphate of magnesium does not give a precipitate, to try the sulphate of calcium reaction before coming to any conclusion. If, in the latter case, we do not obtain an immediate precipitate, then only can we be sure of the absence of the neutral carbonate.—*Journ. de Pharm. et de Chim.*, Series 6, vol. vi., No. 10.

ON THE SOLUBILITY OF AMMONIA IN WATER AT TEMPERATURES BELOW 0° C.

By J. W. MALLET.

ROScoe and Dittmar's table* of the solubility of ammonia in water at different temperatures, under normal pressure, begins at 0° C., and there seems to be no recorded measurements of solubility at any lower temperatures. It is stated on the authority of Fourcroy and Vauquelin† that a "concentrated" aqueous solution of ammonia—the exact amount in solution not given; probably not determined—does not freeze till cooled to between -38° and -41°, that it then forms brilliant flexible needles, and that at -49° it solidifies to a grey gelatinous mass, almost destitute of odour.

It seemed interesting to try the effect of continuing to pass gaseous ammonia into an already strong aqueous solution at temperatures much below zero, and particularly to see whether any visible change of behaviour would mark the presence of enough ammonia to represent the hydroxide of ammonium, often assumed to exist in the ordinary solution, but without any surplus water. The proportion in question—one molecule of ammonia for one of water, might, from an inspection of Roscoe and Dittmar's table, be expected to occur at a temperature but little below zero. Taking advantage of a fall of finely pulverulent dry snow, followed by two or three days of cold weather, during which the atmospheric temperature was about -8° to -12° C., the following experiments were made last winter. In anticipation of such an opportunity, the apparatus had been made ready some time before.

A strong solution of ammonia in water was placed in a burette-like glass tube of the form shown in Fig. 1, of which the larger cylindrical part was about 220 m.m. long and 25 m.m. in internal diameter, the narrow tube below about 100 m.m. long and only about 1 m.m. in internal diameter, with a small glass stopcock about 30 m.m. above the orifice. The cylinder was graduated for about two-thirds of its length into cubic centimetres. By means of an india-rubber stopper, perforated to grasp tightly the narrow tube, and then cut in two in the line of its axis, this part of the burette was fixed in the neck of an inverted gas-jar, the body of which was about 180 m.m. high, with an internal diameter of about 120 m.m., so that the cylindrical body of the burette could be sur-

* *J. Chem. Soc.*, xii., 128—quoted in Roscoe and Schorlemmer's "Treatise on Chemistry," i., 385.

† Gmelin's "Handbook of Chemistry" (Cavendish Soc. transl.), ii., 425.

rounded by a freezing-mixture contained in the jar, without any leakage occurring at the neck, the narrow—almost capillary—tube and stop-cock being below and outside the jar. The open mouth of the burette above was loosely closed by a doubly-perforated stopper, through which passed a small tube for the introduction of ammonia gas, and an alcohol thermometer, each fitting so as to be easily slid up or down. The thermometer was carefully made by Mr. Henry J. Green, of Brooklyn, N.Y., contained pure (uncoloured) alcohol, was graduated on the glass stem, and had the (centigrade) scale verified by comparison with a (Fahrenheit) mercurial thermometer, itself tested by comparison with a gas thermometer, at the following points: $+32^{\circ}$, $+12^{\circ}$, -8° , and -28° F. The gaseous ammonia was obtained by simply heating in a flask the strong aqueous solution. There was, of course, no need for drying it, but it was cooled, and incidentally in large measure dried, by passing it first through an

graduation marks, from which a measured sample might then be taken off. A number of light glass flasks, each of about 300 c.c. capacity, furnished with stoppers, were in advance about three-fourths filled with water and severally weighed with accuracy on a good analytical balance. These flasks of water were brought down to 0° C. by immersion in snow, and—operating out of doors—the several samples of liquid ammonia were run into them from the burette, producing at once such large dilution that there was no further fear of loss of ammonia by standing over, with the stoppers inserted, until the flasks could be re-weighed—the gain of weight in each case representing the amount of liquid taken from the burette—and the amount of real ammonia determined by neutralisation with a standard solution of sulphuric acid. The first sample was taken at -40° C., and then, by simply removing the freezing-mixture of snow and hydrochloric acid from the jar, the temperature of the liquid in

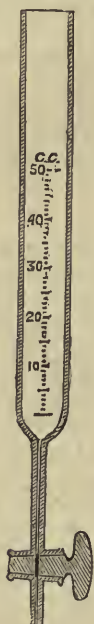


FIG. 1.

empty flask surrounded by snow, and then through a spiral coil of glass tubing immersed in a bath of snow and common salt. Thence by an india-rubber connecting tube it passed into the already strong solution in water contained in the burette. This was surrounded in the jar by a freezing-mixture of snow and commercial hydrochloric acid, both materials having been separately cooled down in advance by snow and salt. In consequence of the evolution of heat as more ammonia was absorbed by the water in the burette, it became necessary to allow ample time for cooling down again, and to renew the freezing-mixture in the jar, drawing off the old mixture by means of a specially arranged and rapidly acting syphon. In order to get the lowest temperature reached (between -45° and -46° C.) it was necessary to doubly cool the last portions of snow and hydrochloric acid, first by snow and salt, and then by separate exposure to some of the already cooled snow and hydrochloric acid mixture. The passage of the gas into the solution was continued until it went through very freely unabsorbed. The liquid in the burette continually increased in volume, so that, when samples were drawn off by the stopcock, enough was first run off to effectually cool the projecting part of the narrow tube and the stop-cock itself, and to bring the level of the remaining liquid down to one of the

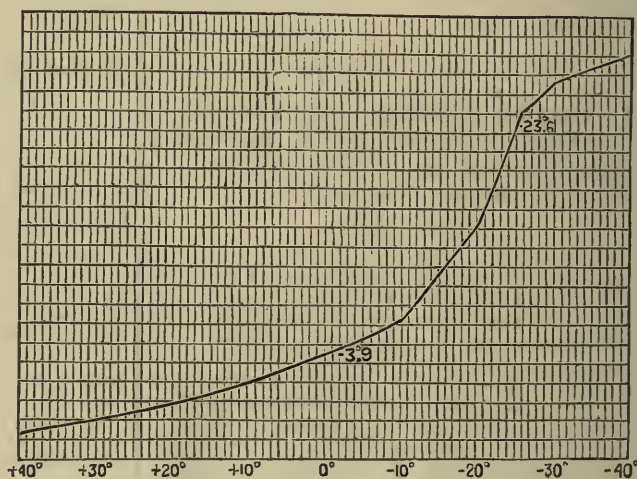


FIG. 2.

the burette was allowed to slowly rise, and as the thermometer marked different desired points it was raised for a moment out of the liquid, another sample drawn off into the water in one of the flasks beneath, and the thermometer lowered again. One or two fairly good measurements of the volumes of the samples were made, but, owing to the necessity for quick work, and the difficulty of seeing satisfactorily through the dimmed surface of the glass, most of these measurements were little to be relied on.

The following results were obtained, so expressed as to form a continuation of Roscoe and Dittmar's table. The atmospheric pressure at the time was 743–744.5 m.m., corrected for temperature of the mercurial column.

At -10° C. 1 grm. of water dissolved 1.115 grms. of ammonia.

At -20° C. 1 grm. of water dissolved 1.768 grms. of ammonia.

At -30° C. 1 grm. of water dissolved 2.781 grms. of ammonia.

At -40° C. 1 grm. of water dissolved 2.946 grms. of ammonia.

The taking up of enough ammonia to form ammonium hydroxide was not attended with any apparent change of behaviour, and continuous liquefaction went on smoothly

to the lowest temperature reached, with no separation of any solid product. After the freezing-mixture had been withdrawn the temperature rose at first very gradually in consequence of rapid surface evaporation of ammonia. At -25°C . (barometer at 743.4 m.m., corr.), steady but not tumultuous ebullition took place and continued for several minutes. A sample taken at this point had the composition: 1 grm. of water and 2.554 grms. of ammonia.

By interpolation from Regnault's results, the tension of the vapour from liquefied ammonia at -25.6°C . would be about 1167 m.m.

A sample was also taken at -3.9° , which proved to consist of 1 grm. of water and 0.947 grm. of ammonia—this last corresponding almost exactly with the calculated proportion for ammonium hydroxide, and agreeing also quite closely with an extension of Roscoe and Dittmar's table to the temperature named, allowance being made for the pressure being a little below normal.

These results are graphically plotted in fig. 2, which includes also the determinations of Roscoe and Dittmar for $+40^{\circ}$ to 0° . It will be seen that the curve changes rapidly a little below the temperature corresponding to the formation of $(\text{NH}_4)\text{OH}$, and the amount of ammonia absorbed is much greater than would be called for by an extension of Roscoe and Dittmar's numbers.

As regards the reversal of curvature before -30° is reached, some allowance must doubtless be made for loss of ammonia by the samples drawn at the lowest temperatures in dropping through the air into the ice-water in the flasks in spite of the precautions taken to minimise loss from this source. And, in view of the fact that liquefied ammonia boils at -33.7° under a pressure of 749.3 m.m. (Bunsen), the amount of ammonia found in the contents of the burette below this point would, of course, go on increasing indefinitely, depending simply on the length of time allowed for condensation and the more or less free supply of the gas. It appears that ammonium hydroxide, if such a substance exist, or the solution of ammonia in water in proportion corresponding to such a compound, continues to dissolve gaseous ammonia, or mixes with liquefied ammonia, down to and beyond the normal boiling-point of the latter, but the proportion dissolved is much greater than would be called for by an extension of the curve representing solubility at temperatures above 0° .

As has been stated, measurement of the volumes of the samples taken could not be very satisfactorily carried out. It may be mentioned, however, that a comparison of volume with weight gave for the liquid at -30° a density of about 0.718, and for that at -40° about 0.731, as referred to water at $+4^{\circ}$. The latter figure being greater than the former may probably be accounted for by the very high value of the coefficient of dilatation for liquefied gases. Calculating from Jolly's results, the density of liquefied ammonia itself at -40° ought to be something like 0.673.—*American Chemical Journal*, xix., No. 9.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING NOVEMBER 30TH, 1897.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolis Water Act, 1871.

London, December 10th, 1897.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 182 samples of water collected by us during the past month, at the several places and on the several days indicated, from the

mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Nov. 1st to Nov. 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 182 samples examined by us, one was recorded as "clear but dull," the remainder being clear, bright, and well filtered.

The rainfall at Oxford shows a further serious deficiency, the actual fall during the month being only 1.15 inches, against a thirty years' average of 2.10 inches, making a deficit of 0.95 inch. The total deficit for the year is now 1.36 inches.

Our bacteriological examinations of 252 samples have given the results recorded in the following table; we have also examined 77 other samples, from special wells, stand-pipes, &c., making a total of 329 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 26 samples) ..	488
New River, filtered (mean of 25 samples) ..	13
Thames, unfiltered (mean of 26 samples) ..	27,173
Thames water, from the clear water wells of five Thames-derived supplies (mean of 122 samples)	27
Ditto ditto highest	218
Ditto ditto lowest	1
River Lea, unfiltered (mean of 26 samples) ..	593
River Lea, from the clear water well of the East London Water Company (mean of 26 samples)	12

The very small rainfall during the past three months has had the usual good effect on the quality of the London water supply, which is now both chemically and bacteriologically excellent.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.
JAMES DEWAR.

A REVISION OF THE ATOMIC WEIGHT OF NICKEL.*

FIRST PAPER.—THE ANALYSIS OF NICKELOUS BROMIDE.

By THEODORE WILLIAM RICHARDS
and
ALLERTON SEWARD CUSHMAN.

(Concluded from p. 296).

WHEN the manipulation had thus been mastered, the art of preparing absolutely pure nickelous bromide had been perfected (see Analysis 7), and the atomic weight of nickel had been approximately determined, the method of procedure in subsequent analyses was changed. The now perfectly clear solution was treated with just enough argentic nitrate, prepared from the purest weighed silver, to complete the precipitation. The mean between the two possible end-points was determined by titrating back.

* Contribution from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, vol. xxxiii., No. 7.

wards and forwards with hundredth normal argentic nitrate and hydrobromic acid solutions (for the details see *Proc. Amer. Acad.*, xxx., 384); and thus was determined the ratio of silver to nickelous bromide entitled Series III. After this end-point had been determined, a slight excess of argentic nitrate was added to the solution, and the whole was violently shaken. The precipitate was collected upon a Gooch crucible, washed with water containing a trace of argentic nitrate, later with pure water, and finally dried and weighed. The traces of asbestos carried away by the wash water were of course determined, and all the usual precautions were taken to insure great accuracy. Thus was obtained the series of results given in Series II.

The Atomic Weight of Nickel.

O = 16.000; Ag = 107.93.

FIRST SERIES (PRELIMINARY).—RATIO = 2AgBr : NiBr₂.

Number of expt.	Sample of NiBr ₂ .	Weight of nickelous bromide in vacuum. Grms.	Weight of insoluble residue. M.grms.	Weight of argentic bromide in vacuum. Grms.	Atomic weight of nickel.
1.	I.	2.26113	3.22	3.88769	58.646
2.	I.	2.80668	7.08	4.82431	58.708
3.	II.	1.41317	3.05	2.42880	58.716
4.	II.	1.71759	0.88	2.95307	58.650
5.	III.	2.48565	5.24	4.27357	58.651
6.	III.	4.32997	15.83	7.44280	58.700
7.	III.	2.18072	0.00	3.74856	58.693

Average.. .. 58.680

SECOND SERIES.—RATIO = 2AgBr : NiBr₂.

Number of expt.	Sample of NiBr ₂ .	Weight of nickelous bromide in vacuum. Grms.	Weight of argentic bromide in vacuum. Grms.	Atomic weight of nickel.
8.	III.	3.28039	5.63892	58.691
9.	III.	2.70044	4.64208	58.686
10.	III.	3.38230	5.81391	58.698
11.	III.	1.33459	2.29435	58.670
12.	IV.	1.25054	2.14963	58.693
13.	IV.	1.32278	2.27384	58.690
14.	IV.	2.24452	3.85805	58.705

Average.. .. 58.690

THIRD SERIES.—RATIO = 2AgBr : NiBr₂.

Number of expt.	Sample of NiBr ₂ .	Weight of nickelous bromide in vacuum. Grms.	Weight of silver in vacuum. Grms.	Atomic weight of nickel.
8.	III.	3.28039	3.23910	58.701
9.	III.	2.70044	2.66636	58.709
10.	III.	3.38230	3.33990	58.689
11.	III.	1.33459	1.31787	58.689
12.	IV.	1.25054	1.23482	58.698
13.	IV.	1.32278	1.30629	58.675
14.	IV.	2.24452	2.21652	58.676

Average.. .. 58.691

In the table above, the first column records the number of the experiment, the second records the number of the sample of nickelous bromide used, while the third records the weight of this salt taken. The extreme right hand column contains the atomic weight of nickel computed from the values contained in the one just to the left of it, and those contained in the third.

A very interesting evidence of the accuracy of these results is the relationship between the amount of silver taken and the amount of argentic bromide obtained. From the second and third series, we find that 15.51556 grms. of nickelous bromide yielded 26.67078 grms. of argentic bromide, requiring 15.32086 grms. of silver. This leads

to the inference that argentic bromide contains 57.444 per cent of silver—a quantity which agrees essentially with the value 57.445 per cent found by Stas. Since the bromine used had been already found to be free from other halogens, and the silver was known to be perfectly pure, we have in these results conclusive proof that no nickel salt was occluded by the argentic bromide, as well as a satisfactory "check" upon the accuracy of the work.

When we examine the results with respect to the various samples of the salt analysed, we find a very interesting and satisfying uniformity. The four samples of nickelous bromide gave the following results for the atomic weight of nickel:—

Sample I..	..	..	58.677
" II..	..	..	58.683
" III..	..	..	58.688
" IV..	..	..	58.689

The slight rise in the value with increasing purity is not large enough to have any weight, for there are analyses in the lowest series giving higher individual results than any in the highest series. Hence we are forced to the conclusion that the least carefully purified specimens of nickelous bromide must have been essentially identical with the most carefully purified. The chances are evidently exceedingly small that the impurities would so combine as exactly to counterbalance one another.

The further discussion of this important question will be reserved until more experimental work has been done. For the present, it is our opinion, at this first halting place in a long investigation, that the atomic weight of nickel cannot be far from 58.69 if O = 16.00, or 58.25 if O = 15.88.

ON THE CHLORONITRIDES OF PHOSPHORUS.*

By H. N. STOKES.

In a former article (*Am. Chem. Journ.*, xvii., 275, 1895; *Ber. d. Chem. Ges.*, xxviii., 437) I have shown that in addition to the phosphonitric chloride, $P_3N_3Cl_6$, discovered by Liebig, there exists another, $P_4N_4Cl_8$, of similar properties, which is formed at the same time, but in smaller quantity. The opinion was expressed that these bodies belong to a series of polymers, $(PNC l_2)_n$, the existence of other members of which was indicated by the formation, in small amount, of a liquid of the same empirical composition (*Am. Chem. Journ.*, xvii., 277, 280, 290). The yield of this secondary product—only 2 per cent of the theoretical or 1 per cent of the pentachloride used—was too small to allow of its preparation in quantities large enough to admit of the isolation of its supposed constituents, but a fractional distillation of the few grms. at my disposal showed that it contained crystalline substances of higher boiling-points than those of the two bodies thus far known.

The method of preparation then employed consisted in distilling phosphorus pentachloride with a large excess of ammonium chloride in a retort, at atmospheric pressure; it offered but little prospect of obtaining the higher members. The total yield of phosphonitric chloride was but 15 per cent of the theoretical, most of the pentachloride being converted into "phospham" by the excess of ammonium chloride, while only those members could be obtained which distil unchanged at atmospheric pressure. Decreasing the amount of ammonium chloride resulted only in a loss of pentachloride by volatilisation, without increasing the yield of the bodies sought after.

* Published by permission of the Director of the United States Geological Survey. From the *American Chemical Journal*, vol. xix., No. 9, November, 1897.

† I propose in future to use the term *phosphorus chloronitride* to denote any body composed of phosphorus, nitrogen, and chlorine, the name *phosphonitric chloride* being reserved for chloronitrides belonging to the series $(PNC l_2)_n$.

The following method has been found to give entirely satisfactory results; several new bodies have been obtained, and the simpler phosphonitric chlorides at least are now easily accessible substances:—If equal molecular weights of phosphorus pentachloride and ammonium chloride be heated in a sealed tube, there results a mixture of chloronitrides, which is partly crystalline and soluble in gasoline, but for the greater part liquid and insoluble in this solvent, and of a high degree of complexity. This may be distilled almost without residue, the distillate being a crystalline mass, impregnated with an oil, and composed almost wholly of a mixture of members of the series $(\text{PNCl}_2)_n$ in nearly theoretical amount, containing about 50 per cent $\text{P}_3\text{N}_3\text{Cl}_6$, and 25 per cent $\text{P}_4\text{N}_4\text{Cl}_8$, the remainder consisting of the higher homologues. From this distillate the new bodies, with one exception, have been isolated.

The series, as at present known, consists of the following (the melting- and boiling-points are corrected):—

	Melting-point.	Boiling-point. 13 m.m.	760 m.m.
Triphosphonitric chloride, $(\text{PNCl}_2)_3$	114°	127°	256.5°*
Tetraphosphonitric chloride, $(\text{PNCl}_2)_4$	123.5°	188°	328.5°†
Pentaphosphonitric chloride, $(\text{PNCl}_2)_5$	40.5-41°	223-224.3°	Polymerises
Hexaphosphonitric chloride, $(\text{PNCl}_2)_6$	91°	261-263°	Polymerises
Heptaphosphonitric chloride, $(\text{PNCl}_2)_7$.			
Liquid at	-18°	289-294°	Polymerises

Polyphosphonitric chloride, $(\text{PNCl}_2)_x$ Below red heat. Depolymerises on distillation.

* 183.8° at 100 m.m.

† 242° at 100 m.m.

There were obtained, further, a liquid residue of the same empirical composition, of a mean molecular weight corresponding nearly to $(\text{PNCl}_2)_{11}$, and a small amount of a chloronitride, $\text{P}_6\text{N}_7\text{Cl}_9$, not belonging to the above series. The absence of the lower members, PNCl_2 and $(\text{PNCl}_2)_2$, is remarkable, and theoretically significant. Indications of a trace of a substance more volatile than the compound $(\text{PNCl}_2)_3$ and of similar but stronger odour, were observed, but there is no evidence that it consists of one of the missing bodies.

One of the most remarkable properties of the phosphonitric chlorides is that each member of the series is converted by heat into the rubber-like *polyphosphonitric chloride*, a body, or mixture of bodies, of very high molecular weight, which is highly elastic and insoluble in all neutral solvents, but which swells enormously in benzene, and which, on distilling at a higher temperature, breaks down into a mixture of all the lower members mentioned above, which can then be separated by appropriate means. In this way it is possible to convert any phosphonitric chloride quantitatively into any other by heat and distillation alone. In preparing any desired member, therefore, we are not limited to the quantity obtained from the first reaction product, but may work the residues over and over again until completely converted into the body sought after. With the exception of a few cases, in which the number of members is limited, as the aldehydes and cyanic acids, this series is therefore unique; I know of no other series of inorganic compounds in which this is possible. Polymerisation takes place slowly, but perceptibly, at 250°, and is almost instantaneous at 350°, while depolymerisation begins at about 350°, and is rapid at a temperature close to incipient red heat. Triphosphonitric chloride, $\text{P}_3\text{N}_3\text{Cl}_6$, is the only member which can be distilled in considerable amount at atmospheric pressure without considerable polymerisation, and even this polymerises almost completely on long boiling; at 760

m.m. pressure the tetra-compound, $\text{P}_4\text{N}_4\text{Cl}_8$, boils at 328.5°, a temperature at which polymerisation occurs quite rapidly, but this, as well as the penta-compound, $\text{P}_5\text{N}_5\text{Cl}_{10}$, and the hexa-compound, $\text{P}_6\text{N}_6\text{Cl}_{12}$, can readily be distilled at 13 m.m.; the hepta-compound, $\text{P}_7\text{N}_7\text{Cl}_{14}$, suffers marked polymerisation on distilling even at this pressure, and its isolation is therefore attended with much loss. Owing to the rapid change at higher temperatures, I have been unable to isolate any of the higher members, which remain as a considerable oily residuum, and there seems to be but little probability of this being effected by any known method, unless by distilling in a nearly absolute vacuum.

The greatest difficulty in the separation of the members is caused by polymerisation. It requires but a small amount of polyphosphonitric chloride to cause the liquid to thicken or gelatinise, and therefore to be incapable of further distillation; and some of this body is always formed in the course of a prolonged fractioning of the higher members. It was found, however, that this polymer is much more easily attacked by water than the lower members; when signs of polymerisation are observed, it is only necessary to interrupt the distillation and heat the residue for some time with water, when the resulting oil is again in a condition to continue fractioning.

The loss in this operation is small, but the tediousness of a fractional distillation is thereby extraordinarily increased.

It is noteworthy that no regular progression exists in the melting-points of the phosphonitric chlorides, and the same is true of their solubility in the ordinary neutral solvents, but the solubility varies in the same sense as the fusibility. Of the members of known molecular weight, the second, tetraphosphonitric chloride, is the least soluble and has the highest melting-point, while the corresponding tetrametaphosphoric acid is the least soluble and most stable of the derived acids. With respect to their stability towards water, the new members (polyphosphonitric chloride excepted) resemble those already described, being scarcely attacked by prolonged boiling. In ethereal solution, however, there is a perceptible decrease of stability towards water as we rise in the series—a fact already noted with regard to the first two members (*Am. Chem. Journ.*, xvii., 289).

Notwithstanding the high molecular weight of the bodies isolated, no indication of isomers has been observed, although the fractioning was carried out very thoroughly up to 300° at 13 m.m.

The investigation will be continued with the object of obtaining the metaphosphoric acids, and, if possible, the two missing lowest members of the phosphonitric chloride series. The right of further investigation in this field, however, is not reserved.

Experimental Part.

A mixture (which need not be very intimate) of 4 parts perfectly dry phosphorus pentachloride and 1 part ammonium chloride, as required by the equation $\text{PCl}_5 + \text{NH}_4\text{Cl} = \text{PNCl}_2 + 4\text{HCl}$, is introduced into an ordinary "bomb" tube, which has previously been drawn out to a neck. It is practicable to fill the tube entirely to the neck, so that the charge for a tube of ordinary dimensions is about 125 grms., yielding 50–55 grms. of chloronitrides. After sealing, the length of the neck, exclusive of the rather long capillary, should be about 10 c.m. As the mixture liberates 55 per cent hydrochloric acid, it is necessary to regulate the heating with great care and to open the tube repeatedly. The temperature of the furnace is allowed to rise to 150°, at which the reaction begins, when the gas is at once shut off, and the tube opened at about 100° (in the furnace!). This operation is repeated several times, the temperature being allowed to rise 1–2° higher each time. When the evolution of hydrochloric acid has slackened and the contents of the tube are mainly liquid while hot, the temperature may be carried to 200° or higher, until little

or no gas is given off. The operation requires care and judgment, but with careful working it is possible to avoid explosions, and to obtain with a four-tube furnace about 200 grms. of mixed chloronitrides in sixteen hours.

The contents of the tube, after cooling, generally consist of a buttery mass or of a thick yellow liquid filled with fine prisms and plates; if heated much above 200°, the liquid frequently separates into two layers. The crystals are soluble in gasoline, but the bulk of the product remains as an immiscible oil.

The neck of the tube is now bent down, the tube placed in an inclined combustion furnace, and by cautious heating, finally to incipient redness, the contents are distilled out. There remains in the tube a very voluminous spongy black residue, of inconsiderable weight, due to unavoidable impurities, and to the impossibility of causing complete reaction in the sense of the above equation. The distillate consists of a crystalline mass impregnated with a yellow oil, and contains about 95 per cent of the theoretical amount of phosphonitrilic chlorides, with some phosphorus pentachloride, the chloronitride $P_6N_7Cl_9$, and other substances of unknown nature. Before proceeding further, it is necessary to remove the pentachloride, and for this purpose the distillate is melted, poured into cold water, and the flask heated in the water-bath for about two hours, the liquids being mixed by blowing air through them. The chloronitrides are then allowed to clear under the hot water, and forced out by means of a wash-bottle arrangement; a separatory funnel cannot be used, as the substance solidifies in the neck, and if allowed to solidify under the wash water it absorbs so much of this as to cause annoyance in the subsequent distillation. Special drying before distilling is unnecessary.

The product is then distilled up to 200° at 13 to 15 m.m., using an Anschütz flask, as the distillate solidifies instantly on cooling. The residue, containing the members $P_5N_5Cl_{10}$ up, is set aside for later systematic fractional distillation.

(This residue contains the small amount of $P_6N_7Cl_9$, formed as a secondary product of the original reagents, and as this is apt to cause inconvenience at a later stage, by accumulating with the $P_6N_6Cl_{12}$, it is perhaps well to remove as much as possible at this point. For this purpose the residue is allowed to stand for a day or two at the room temperature, and the crystals removed by sucking out under a good vacuum, best in a large Gooch crucible. The filtrate is cooled for a day or two in a refrigerator, and the new crop of crystals separated in the same way, the filtering flask being allowed to stand in the ice-box. The oily filtrate is set aside, and the united crystalline products distilled up to 240° at 13 m.m., whereby most of the $P_5N_5Cl_{10}$ passes over. The residue, consisting of $P_6N_6Cl_{12}$, the small amount of $P_6N_7Cl_9$, and the adhering oil, is allowed to crystallise in the refrigerator, and the viscous mass is extracted several times with small amounts of gasoline (boiling at 50° to 80°). The residue is boiled with benzene, which extracts the $P_6N_7Cl_2$, which crystallises on concentrating and cooling. The portion dissolved by the gasoline is worked up with the other residues. This is the method actually employed, but I am not entirely convinced of its necessity, as it is not possible to remove all the $P_6N_7Cl_9$ in this way).

The distillate, about 70 per cent, consists essentially of $P_3N_3Cl_6$ and $P_4N_4Cl_8$, which, if desired, may be easily separated by fractional distillation *in vacuo*, followed by crystallisation from benzene. This is more convenient than the method of separating by steam (*Amer. Chem. Journ.*, xvii., 280). If it is desired to convert it into the higher members, it is placed in a combustion tube bent down at about 20 c.m. from the open end, and which it should not fill more than one-half after melting. This is laid in an inclined combustion furnace, and heated to gentle boiling of the contents. It is well to heat the tube somewhat strongly at a short distance above the liquid, as

superheating the vapour promotes polymerisation. The time required for polymerisation varies greatly; pure triphosphonitrilic chloride may require two hours or more; with the above mixture the time is less, and is shorter the higher the boiling-point; it is shortened by adding already gelatinised substance, which causes the liquid to thicken, and may then be but a few minutes; it is also shortened by heating under pressure. Sooner or later the liquid begins to thicken, and finally it is converted into a stiff, transparent mass, with little or no liquid, and generally discoloured by traces of organic matter. The tube is then connected with a long-necked receiver, exhausted, and the depolymerisation and distillation effected by heating, from the front backward, to incipient redness. This part of the operation proceeds rapidly, as it is only necessary to guard against frothing over, and to ensure complete condensation, the latter being easily effected by having the limb of the tube at least 20 c.m. long: 100 grms. can be worked up at one time, and the tube can be used repeatedly. The residue does not weigh more than a few m.grms. The distillation may also be made at atmospheric pressure, but the yield of higher products is thereby diminished. The distillate, which entirely resembles that first obtained, except in containing no phosphorus pentachloride and no $P_6N_7Cl_9$, is distilled as before, the washing being omitted. In this way the whole quantity of material can finally be converted into a mixture of members higher than $P_4N_4Cl_8$.

The united residues boiling above 200° are now submitted to systematic fractional distillation at 13 to 15 m.m., using an Anschütz flask, provided with a "trap," to prevent flowing back. During the first distillation polymerisation generally begins when the temperature of the bath has reached 270°, but with later distillations at a higher temperature, and the higher the purer the fractions are. When polymerisation begins, which is indicated by frothing and thickening, the operation is interrupted, and the residue heated in the flask with water in the water-bath until it has completely liquefied, which is assisted by agitation, the oil separated,* and the distillation continued. It has not been found practicable to continue the distillation at a higher temperature than that obtained by heating the bath to 370°, for the liquid begins to polymerise in a few moments, and but an inconsiderable distillate can be obtained. Moreover, at this temperature the polymer shows signs of breaking down into simpler bodies, and the distillate does not consist only of high-boiling members. The total amount of final residue is not very great, and as shown below consists likewise of phosphonitrilic chlorides of still higher molecular weight. In later distillations, from 200° upward, polymerisation usually stops the process at 260° to 270°, but after appropriate washing the residue may be distilled to a much higher temperature. After 8 to 10 distillations three main fractions are obtained, which are then worked up separately. As $P_5N_5Cl_{10}$, though crystalline, is extremely soluble, it is necessary to carry out the distillations with the first main fraction until a practically sharp boiling-point is obtained, in which connection it may be noted that at 17 to 20 m.m. a change of 1 m.m. pressure causes a change of about 1° in the boiling-point, and at 13 m.m. a change of about 2°. $P_7N_7Cl_{14}$, being liquid, must also be isolated by distillation only, but at its boiling-point polymerisation is so rapid that great loss ensues during a series of distillations. The final purification of $P_6N_6Cl_{12}$ can be effected by repeated re-crystallisation from benzene, combined with treatment with gasoline to remove the $P_6N_7Cl_9$, which always accompanies it, having nearly the same boiling-point: a complete separation of these two bodies can scarcely be effected by distillation alone.

Owing to many modifications introduced in developing the above method, no accurate statement of the yield can be given; the final product was about 225 grms.

*In this case a separatory funnel may be used, as the higher chloronitrides are liquid below 80°.

$P_5N_5Cl_{10}$, 110 grms. $P_6N_6Cl_{12}$, 10 grms. $P_7N_7Cl_{14}$, and 5 grms. $P_6N_7Cl_9$.

Analytical Methods.

With the exception of polyphosphonitric chloride, the chloronitrides were analysed by decomposing in the following manner:—

For phosphorus, by warming with alcohol and a little ammonia in a platinum crucible until completely dissolved, evaporating to dryness, and heating to fuming for an hour with strong sulphuric acid, the crucible being kept covered.

For nitrogen, by treating as above, omitting the ammonia.

For chlorine, by heating with alcohol and ammonia. It is necessary to precipitate with silver nitrate in the presence of a large volume of 10 per cent nitric acid and to filter hot, in order to avoid the formation of silver metaphosphimides, which are difficultly soluble in dilute nitric acid.

In decomposing polyphosphonitric chloride, which is attacked by water alone, the alcohol was omitted. The method of Carius was used for determining chlorine, as it was found that otherwise compounds insoluble in dilute nitric acid were formed. For the other chloronitrides this method offers no advantage.

Molecular weight determinations were made by the boiling-point method with the apparatus of Hite (*Amer. Chem. Journ.*, xvii., 512), using as solvent carefully purified and dried benzene.

(The molecular weight of $P_3N_3Cl_6$ has been determined by the vapour density method (*J. Chem. Soc.*, [2], ii., 225; *Amer. Chem. Journ.*, xvii., 283). A series of determinations by the boiling-point method gave 346, 350, 353. Calculated, 347.9).

Pentaphosphonitric chloride, $P_5N_5Cl_{10}$.—This body, carefully purified by fractional distillation, as described above, gave on analysis:—

	Calculated for $P_5N_5Cl_{10}$	Found.
P	26.75	26.87
N	12.11	12.05
Cl	61.14	61.42

Ratio, P : N : Cl = 1 : 0.99 : 2.00.

Molecular Weight. Solvent: Benzene.

Grms. solvent.	Grms. substance.	Elevation.	Molecular weight found.	Percentage variation from theoretical.
46.49	1.4688	0.137°	619	+6.2
"	2.9439	0.287°	589	+1.6
"	4.4337	0.437°	583	+0.5

Calculated for $P_5N_5Cl_{10}$, 579.8.

Pentaphosphonitric chloride fuses at 40.5°—41°, and boils at 223°—224.3° (corr.) at 13 m.m. Its vapour is without the pronounced and characteristic aromatic odour possessed by that of triphosphonitric chloride. At its melting-point it is miscible in all proportions with benzene, gasoline, ether, and carbon disulphide, and cannot be re-crystallised from any of these solvents, in fact, small fragments liquefy instantly in their concentrated vapours. Glacial acetic acid also dissolves it quite readily, and from this solution water throws it out as an oil, solidifying at once on touching. It shows a decided tendency to superfusion, especially when not absolutely pure; when left by evaporating its ether or benzene solution, it may remain liquid for days, but solidifies at once on touching with a glass rod, usually to a decidedly crystalline mass, at other times to a transparent glass. The pure substance, when fused, slowly solidifies, long flat crystals shooting out through the liquid, which are limited only by the size of the vessel, crystals of 10 c.m. in length being readily obtained. It contracts greatly on solidifying. When pure the solidified mass is naturally dry, but the least contamination with other members of the series causes a portion to remain liquid, which is easily detected by crushing on a piece of filter-paper; this is a very good

test of its purity. This tendency to superfusion must be borne in mind in separating it by fractional distillation; a nearly pure sample will remain liquid much longer than the higher or lower fractions. In ether solution it is perceptibly more easily attacked by water than the preceding chloronitride, but hot water alone is almost without action.

Hexaphosphonitric Chloride, $P_6N_6Cl_{12}$.—After repeated crystallisation from benzene,* this gave—

	Calculated for $P_6N_6Cl_{12}$	Found.
P	26.75	26.98
N	12.11	12.37
Cl	61.14	60.98

Ratio, P : N : Cl = 1 : 1.01 : 1.98.

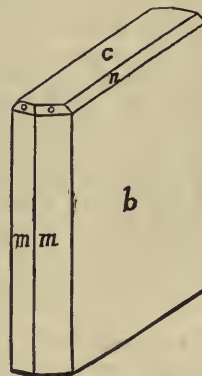
Molecular Weight. Solvent: Benzene.

Grms. solvent.	Grms. substance.	Elevation.	Molecular weight.	Percentage variation from theoretical.
47.12	1.8058	0.152°	673	-3.2
"	3.6190	0.293°	700	+0.6
45.72	1.0101	0.083°	711	+2.1
"	4.8998	0.365°	718	+3.2
"	8.0070	0.664°	704	+1.2

Calculated for $P_6N_6Cl_{12}$, 695.8.

Hexaphosphonitric chloride fuses at 91° (corr.), and boils at 261°—263° (corr.) at 13 m.m., and at 281°—282° (corr.) at 26 m.m. It may be re-crystallised from benzene, in which, however, it is more soluble than triphosphonitric chloride; ether, gasoline, and carbon disulphide also dissolve it readily; in alcohol it dissolves somewhat slowly with decomposition. It shows no tendency to superfusion. It crystallises well, in rather large crystals, which were examined by Mr. Wirt Tassin, to whom I am indebted for the following statement:—

" $P_6N_6Cl_{12}$ crystallises in the orthorhombic system in long prismatic crystals, showing the following forms:—*c* (001), *b* (010), *m* (110), *o* (111), *n* (011). Of these *c* is the dominant form; *b* large and well developed; *m* fair, though usually narrow; and *o* and *n* small and usually in



similar development. *c*, *b*, *m*, *o* is the combination occurring most frequently; less often *c*, *b*, *m*, *n*, *o*; and rarely *c*, *b*, *m*. Angles *m* 57° 28', *b* : *m* 61° 16', *b* : *n* 40° 23', *n* : *c* 49° 37'. Axial ratio *a* : *b* : *c* = 0.54824 : 1 : 1.17568. The crystals are optically positive. Plane of the optic axes (100); colourless to white; transparent; and have a perfect basal cleavage."

It is scarcely attacked by boiling water, but if kept in moist air it very slowly evolves hydrochloric acid. Its ether solution, shaken with water, slowly gives a metaphosphimic acid; syrupy chlorhydrines are formed as intermediate products.

(To be continued).

* A contamination with $P_5N_5Cl_{10}$ may be detected by treatment with gasoline, when the much smaller crystals of the latter are seen to dissolve much more slowly.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, December 2nd, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

CERTIFICATES were read for the first time in favour of Messrs. Charles Edward Britain, B.Sc., 11, Highfield, Scarborough; William Arthur Caldecott, B.A., Box 1891, Johannesburg, S.A.R.; John Cooper, B.Sc., 20, Derwentwater Road, Gateshead; Frederick Cowling, Clay Cross, near Chesterfield; Wilbraham T. A. Edwards, Reduit, Mauritius; Frederick Gilderdale, 3, Havelock Street, Newcastle; William Setten Gilles, Coniston, Cedars Road, Beckenham; William Hobson Mills, B.A., Jesus College, Cambridge; Frank Forster Renwick, Glengall, Woodford Green, Essex; William Colebrook Reynolds, 64, Lydford Road, Paddington, W.; Andrew Jamieson Walker, B.A., Killycadden, Killygordon, Co. Donegal; Ernest Charles Weismüller, 30, Pepys Road S., New Cross, S.E.

The following were duly elected Fellows of the Society:—John Ball, Ph.D.; William Ball; Alec. Alfred Beadle; Richard Oxley Burland, J.P.; Alexander McLean Cameron; Alexander Clarkson; Frank Collingridge, B.Sc.; James Murray Crofts, B.A.; John Daniell; Andrew James Dixon, F.I.C.; Oscar Guttmann, F.I.C.; Robert Hamilton; John Harger, B.Sc., Ph.D.; James Walter Horseman; Charles Kelly; Tom Lemmey, B.A.; James Scott MacLaurin, D.Sc.; Alan Macmullen, B.A.; Charles Jodrell Mansford, B.A.; Edward Masters; John A. Mathews, M.A. M.Sc.; Philip George Gregory Moon; James Charles Philip, B.Sc., Ph.D.; Alexander Ferguson Reid; Ernest Henry Roberts; Edward Sydney Simpson, B.E.; Robert Francis Wood Smith; Thomas Southern, Jun.; Frederick William Steel; Michael Edmund Stephens; George Stubbs; Edward Howard Tripp, Ph.D.; John Scriven Turner; Framjee Khursedjee Viccajee; Percy John Vinter, M.A.; Arthur James While; Francis Samuel Young, M.A.

The PRESIDENT called the attention of Fellows to the fact that, although according to the Council regulations no paper could be announced which had not been received, it was open to any Fellow to make a communication at a meeting of the Society in the event of there being available time, provided that he handed a written statement to the Secretaries of the essence of his communication.

In answer to a question from Dr. Hake, the PRESIDENT said that the appearance of such communications in the *Proceedings* would be subject to the editorial discretion of the Secretaries, and that when presented as full papers they would come before the Publication Committee in the usual way.

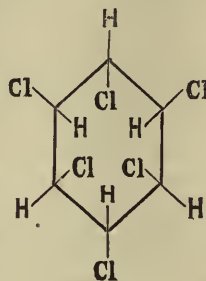
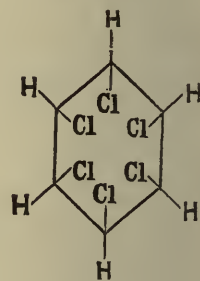
The following papers were read:—

*124. "The Representation of the Isomeric Benzene Hexachlorides by Collie's Space-formula." By FRANCIS EDWARD MATTHEWS, Ph.D.

This paper discusses Collie's space-formula for benzene from the point of view of the halogen hex-addition compounds. The formula is shown to explain the existence of two isomeric hexachlorides satisfactorily, and the accompanying formulæ are proposed for these compounds.

These formulæ likewise explain the differences in stability towards alcoholic alkalis. The α -substance, containing the chlorine in the ortho-position to, and on the same side of the carbon nucleus as the hydrogen atoms with which it is removed in the form of hydrogen chloride, is readily decomposed; the β -substance, in which the hydrogen and chlorine are on opposite sides of the nucleus, has much greater stability. The remainder of the paper discusses the formation of benzene di-derivatives from mono-derivatives, and it is maintained that their

formation is best explained by assuming the previous formation of unstable *ortho*-addition compounds instead of unstable *meta*-compounds, as Collie has suggested.

 α -Compound. β -Compound.

DISCUSSION.

Dr. WYNNE asked whether any explanation could be given of the formation of 1 : 2 : 4-trichlorobenzene from the benzene hexachlorides by the action of alcoholic potash. The usually accepted formulæ for benzene gave no clue to the reason for the production of unsymmetrical derivatives in such cases, and he was unable to see that the formulæ now proposed were more satisfactory in this respect.

Dr. LAPWORTH pointed out that the author's statement as to the impossibility of explaining the production of two different hexachlorides from benzene by means of any formula prior to Collie's, appeared to need qualification. It is easily seen that Kekulé's formula represents several stereoisomeric substances, as each "doubly bound" pair of carbon atoms may form a fumaroid or maleoid combination. Taking into consideration the circumstance that, for a number of reasons, the formula must be considered as a labile one, the positions of the ethylenic and single linkings being supposed to alternate, it is not impossible that, under the conditions of interaction of chlorine and benzene, the latter may react in the form of two or more of its possible stereoisomers. The addition of chlorine to a pair of carbon atoms double bound as in fumaroid compounds would, of course, afford a trans-dichloro-derivative, whilst a cis-dichloro-compound would result if the original combination were of the maleoid type. Kekulé's formula, therefore, would appear to afford a perfectly satisfactory explanation of the production of more than one benzene hexachloride.

Mr. E. J. PARRY asked if Dr. Matthews had any experimental evidence for assigning the symmetrical formula to the α -hexachloride, and the unsymmetrical to the β compound. If the explanations of substitution offered by Collie and the author were correct, there should certainly be twice as much *ortho*- as *para*-dichlorobenzene produced when chlorobenzene is chlorinated. Further, he could not understand why, if these explanations were correct, the nitration of chlorobenzene should give *ortho*- and *para*-compounds whilst the chlorination of nitrobenzene, or the nitration of nitrobenzene, should yield *meta*-compounds.

Dr. MATTHEWS, in reply, stated that it had always seemed to him a remarkable fact that 1 : 2 : 4-trichlorobenzene alone was produced by the action of alkalis upon the benzene hexachlorides. He had made several attempts with large quantities of material to isolate the symmetrical modification, but always without result. The formation of the 1 : 2 : 4-compound could, however, be easily explained, either by Kekulé's or Collie's formula.

With regard to Dr. Lapworth's remarks, the formulæ proposed above account for the production of two and not of a greater number of isomerides which might be expected if these hexachlorides were regarded as *cis*- and *trans*-modifications; whilst certain properties of the compounds do not seem to harmonise with the idea that they are stereoisomerides.

The answer to Mr. Parry's questions are contained in the paper itself; the difference in stability of the two hexachlorides towards alkalis is explained by assuming that hydrogen chloride is more readily removed from atoms in the ortho-position and on the same side of the carbon nucleus than from those in which these conditions do not obtain. Hence the above formulæ were assigned.

*125. "Compounds of Piperidine with Phenols." By OTTO ROSENHEIM, Ph.D., and PHILIP SCHIDROWITZ, Ph.D.

With a view of obtaining substances of the general formula $(C_6H_{6-n})(C_5H_{10-n})_n$, which seemed to be of interest on account of their relation to the phenylenediamines and polyamines, the authors studied the action of piperidine on phenols and their derivatives in the presence of dehydrating agents. Although so far unsuccessful in this direction, a series of addition products in the nature of salts was observed, in which piperidine acts as the base and the phenol as the acid. They are well crystallised compounds, easily obtained by the interaction of their components, usually in ethereal solution. They are resolved into their constituents by strong acid or alkalis. M. Oechsner de Coninck (C. R., 1897, cxxiv., 563) describes a number of colour reactions obtained by the action of piperidine and other bases on phenols in dilute aqueous solution, but has apparently not observed the formation of the addition compounds described in the paper.

The influence of the number and position of the oxy- and nitro-groups in the phenols on the additive capacity of the piperidine molecule was studied, but no general rule could be deduced.

The following compounds were analysed, and are described in the paper:—Compounds of piperidine (1 mol.) with pyrocatechol (2 mols.), guaiacol (2 mols.), hydroquinone (1 mol.), pyrogallol (1 mol.), vanillin (1 mol.), *o*- and *p*-nitrophenol (1 mol.), picric acid (1 mol.), 1:2:4-dinitrophenol (1 mol.). Phenol, *p*-chlorophenol, resorcinol, phloroglucinol, *m*-nitrophenol, and α - and β -naphthol did not furnish crystalline compounds.

EDINBURGH UNIVERSITY CHEMICAL SOCIETY.

Monday, November 29th, 1897.

Mr. W. W. TAYLOR, M.A., B.Sc., in the Chair.

DR. BOLAM read a paper on "Electrolysis in Organic Chemistry."

The paper was mainly historical, and dealt first with the work of Kolbe, Bourgoin, and Kekulé. It was pointed out that Löb had recently given a confirmation of Kekulé's "anhydride theory" by electrolysis phthalic acid.

When phthalic acid is dissolved in alcohol and a few drops of an acid added to increase the conductivity, the passage of a weak current through the solution for some time gives an almost quantitative yield of phthalic anhydride. Longer duration of the current results in the formation of phthalic ether in large quantity.

Dr. Bolam also referred to the use of the alternate current, and concluded by referring to some technical applications in organic chemistry.

There was a large attendance, and Dr. Bolam was thanked for his paper.

CORRESPONDENCE.

ZINC IN WATER.

To the Editor of the Chemical News.

SIR,—I read in the CHEMICAL NEWS (vol. lxxvii., p. 293) that Mr. Percy Richards has detected zinc in a sample of

water used for drinking purposes, in Berkshire, which was supplied by a galvanised iron pipe.

It may be interesting to note that among the samples of water examined last year in my laboratory there were some from the island of Madeira which presented the same character, and which had, likewise, been conducted into the town of Funchal by galvanised iron pipes. There was a large deposit of oxide and carbonate of zinc in one of the bottles.

Putting aside the zinc contamination, the water was of great purity. As zinc is a metal whose compounds have a noxious action upon the economy, it is evident that galvanised iron pipe cannot be used with safety to supply water for drinking.—I am, &c.,

T. L. PHIPSON, Ph.D.

Casa Mia, Putney, S.W.,
December 18, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société Chimique de Paris.

Series 3, Vol. xvii.-xviii., Nos. 16-17.

Action of Chlorine on Pentachlorethane in the presence of Chloride of Aluminium.—A. Mouneyrat.—Although chloride of aluminium has a special action on chloral (removing oxygen), it seemed to the author to be of interest to verify whether or not this powerful synthetic agent might not be a chloridising agent in the acyclic series. He therefore placed 150 grms. of pentachlorethane (C_2Cl_5H) in a flask heated to 70° and provided with a vertical condenser, and exposed to diffused light. Dry chlorine was passed through for thirty hours. This gas was not absorbed, no hydrochloric acid gas was given off, neither was there any hexachlorethane formed. He then added about 30 grms. of anhydrous chloride of aluminium, and again passed a current of dry chlorine as before. This time the gas was completely absorbed, and HCl was given off. After four hours the contents of the flask were solid. On throwing small portions of this white mass into water, hydrochloric acid is given off, and a white powder rises to the surface. This powder was dissolved in ether, the solution dried over chloride of calcium, and distilled. A body smelling strongly of camphor was obtained, sublimating at the ordinary temperature. Analysis showed that it was hexachlorethane (C_2Cl_6), which was formed according to the following equation: $-CCl_3, CCl_2H + Cl_2 = CCl_3, CCl_3 + HCl$. It is an excellent method of preparing hexachlorethane, and the return is almost theoretically exact.

Action of Chlorine on Tetrabromide of Acetylene in the presence of Chloride of Aluminium.—A. Mouneyrat.—Thirty grms. of anhydrous chloride of aluminium were added to 100 grms. of tetrabromide of acetylene, in a flask furnished with a vertical condenser and heated to 70 – 80° ; hydrochloric acid is strongly given off. After four hours, a current of dry chlorine was passed, and at first more hydrochloric acid was given off, followed by torrents of bromine. When the latter ceased and no more chlorine was absorbed, the mass was thrown, a little at a time, into water containing soda-lye; a white powder came to the surface if the temperature had not exceeded 80° , and if the quantity of chloride of aluminium added was small; if, on the other hand, the temperature had exceeded 80° , and if the quantity of chloride of aluminium was in excess, the powder is black. After rectification, analysis showed this powder to be hexachlorethane, C_2Cl_6 , formed according to the following equation: $CHBr_2, CHBr_2 + Cl_2 = CCl_3, CCl_3 + Br_2 + (HCl)_2$.

On Glucosines.—C. Tanret.—The author finds that by acting on glucose by ammonia at 100° a series of bases is formed, among which are the glucosines α and β . MM. Brander and Stoeckel contest the formulæ of these glucosines, maintaining that the glucosine α , boiling at 136° , has not the formula $C_6H_5N_2$, but is a mixture of pyridine, pyrazine, and methylpyrazine. As for the glucosine β , boiling at $155-160^{\circ}$, these authors only obtained a few decigrams. of dimethylpyrazine boiling at $147-155^{\circ}$. M. Tanret shows that these conclusions are incorrect, and that the formulæ he gave are the true ones.

Hydrochlorate of Glucosamine.—C. Tanret.—The hydrochlorate of glucosamine possesses bi-rotation, but not always to the same extent. The author imagined that there must be modifications of this body, and he found that hydrochlorate of glucosamine- α had a rotatory power of $\alpha_D = +100^{\circ}$; and that the modification β , in a solution of 1 part in 2.5 parts of water gave $\alpha_D = 77.50^{\circ}$. The two modifications of hydrochlorate of glucosamine have a distinct rotatory power; and they, moreover, crystallise in two different systems.

On Active Methylbutylenediamine (Methyl-2, $NH_2CH_2, CH, CH_2, CH_2NH_2$ Diaminobutane 1.4),

—L. Etaix and P. Freundler.—The authors find that the successive operations to which they submitted the primitive body have not racemised it to any noticeable degree, though it might have been expected that either the action of hydrazine or of hydrochloric acid at 150° would destroy its rotatory power. It would appear from this that racemisation depends, not on the agents of transformation, but on the groupings which are attached to the asymmetric carbon.

On Dinitrophenyl-diacetyl-methane.—F. Mottelet.—Acetylacetate of sodium is dissolved in strong alcohol in a water-bath, and to this solution, warm, is added an equal quantity of chlordinitrobenzene. A white crystalline deposit is immediately formed. When this deposit no longer increases the reaction is considered to have ended. The mass is thrown into water; an oil is deposited; this is acidulated slightly with hydrochloric acid, and the oil crystallises. The crystals are found by analysis to consist of dinitrophenyl-diacetyl-methane.

Action of Chloride of Ethyloxalyl on Diphenyl in the presence of Chloride of Aluminium.—L. Rousset.—If small portions equal to one molecule of chloride of ethyloxalyl are dropped into a boiling sulphocarbonic solution containing one molecule of diphenyl and 133 grms. of chloride of aluminium, the theoretical quantity of hydrochloric acid is given off, and a brown mass, insoluble in sulphide of carbon, is left; this is treated with water, the sulphocarbonic layer is washed with water containing HCl, and then with water; the sulphide of carbon is driven off, and the residue purified in vacuo. It answers to the formula $C_6H_5, C_6H_4, CO, COOC_2H_5$.

Action of Chloride of Ethyl-oxalyl on Ethyl- α -naphthol in the presence of Chloride of Aluminium.—L. Rousset.—The condensation of chloride of ethyl-oxalyl with ethyl- α -naphthol is carried out in the same way as has previously been described for methyl- α -naphthol. The α -ethyloxynaphthylglyoxylic ether thus obtained boils at 240° to 245° under a pressure of 10 m.m. The glyoxylic acid which results from its saponification, by means of soda in aqueous solution, melts at 160° , and gives with aniline a phenylimide which crystallises in benzene in small yellow grains, fusible at 72° .

On the Perkin Reaction applied to some Aldehyds of the Naphthalene Series.—L. Rousset.—Not suitable for abstraction.

Products of Condensation of Saccharine with the Phenols.—P. Sisley.—Saccharine and resorcin heated together with sulphuric acid gives a yellowish brown

viscous mass. After cooling and throwing into water, yellow-brown crystals are formed; they are soluble in alcohol and acetic acid, but not in ether or chloroform, and they do not contain nitrogen. Its alkaline salts are very soluble, and possess a beautiful green dichroism; they dye silk in the same way as uranine. Analysis shows it to be identical with the sulphurine resorcin of Remsen. When treated with bromine in alcoholic solution it easily gives a bromised derivative forming red crystals, slightly soluble in water, and soluble in the alkalis, forming a red solution with a yellow fluorescence.

On Veratrylene-diamine.—Cl. Moureu.—Not suitable for abstraction.

Estimation of Phosphoric Acid.—H. Lasne.—Already inserted in full.

Apparatus for the Commercial Analysis of Gas.—L. Vignon.—A long paper, not suitable for abstraction.

Estimation of Small Quantities of Methyl-alcohol, Formic Aldehyd, and Formic Acid.—M. Nicloux.—Already inserted.

Cryoscopy of Milk.—A. Ponsot.—Hamburger found a variation of 0.013° for the congealing point of milk, with a mean value of -0.561° , his results agreeing with those obtained by Winter; but when working with a Beckmann apparatus he found an average of -0.520° (-0.512° to -0.529°). A large part of this difference must be due to defective working,—for instance, he uses such a degree of cold that the milk, when introduced into the cryoscopic tube, is brought to a condition of surfusion. It is necessary that the same method should be followed by all, in order to get comparative results.

MEETINGS FOR THE WEEK.

TUESDAY, December 28th. } Royal Institution, 3. (Christmas Lec-
THURSDAY, December 30th. } tures). "Principles of the Electric
SATURDAY, Jan. 1st, 1898 } Telegraph," by Prof. Oliver Lodge.

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THE CHEMICAL NEWS.

VOL. LXXVI., No. 1988.

ON THE DENSITIES OF CARBONIC OXIDE, CARBONIC ANHYDRIDE, AND NITROUS OXIDE.*

By Lord RAYLEIGH, F.R.S.

THE observations here recorded were carried on by the method and with the apparatus described in a former paper ("On the Densities of the Principal Gases," *Roy. Soc. Proc.*, liii., p. 134, 1893), to which reference must be made for details. It must suffice to say that the globe containing the gas to be weighed was filled at 0° C., and to a pressure determined by a manometric gauge. This pressure, nearly atmospheric, is slightly variable with temperature on account of the expansion of the mercury and iron involved. The actually observed weights are corrected so as to correspond with a temperature of 15° C. of the gauge, as well as for the errors in the platinum and brass weights employed. In the present as well as in the former experiments I have been ably assisted by Mr. George Gordon.

Carbonic Oxide.

This gas was prepared by three methods. In the first method a flask, sealed to the rest of the apparatus, was charged with 80 grms. re-crystallised ferrocyanide of potassium and 360 c.c. strong sulphuric acid. The generation of gas could be started by the application of heat, and with care it could be checked and finally stopped by the removal of the flame with subsequent application, if necessary, of wet cotton-wool to the exterior of the flask. In this way one charge could be utilised with great advantage for several fillings. On leaving the flask the gas was passed through a bubbler containing potash solution (convenient as allowing the rate of production to be more easily estimated), and thence through tubes charged with fragments of potash and phosphoric anhydride, all connected by sealing. When possible, the weight of the globe full was compared with the mean of the preceding and following weights *empty*. Four experiments were made with results agreeing to within a few tenths of a m.grm.

In the second set of experiments the flask was charged with 100 grms. of oxalic acid and 500 c.c. strong sulphuric acid. To absorb the large quantity of CO₂ simultaneously evolved a plentiful supply of alkali was required. A wash-bottle and a long nearly horizontal tube contained strong alkaline solution, and these were followed by the tubes containing solid potash and phosphoric anhydride as before.

For the experiments of the third set *oxalic acid* was replaced by *formic*, which is more convenient as not entailing the absorption of large volumes of CO₂. In this case the charge consisted of 50 grms. formate of soda, 300 c.c. strong sulphuric acid, and 150 c.c. distilled water. The water is necessary in order to prevent action in the cold, and the amount requires to be somewhat carefully adjusted. As purifiers, the long horizontal bubbler was retained, and the tubes charged with solid potash and phosphoric anhydride. In this set there were four concordant experiments. The immediate results stand thus:—

Carbonic Oxide.

From ferrocyanide	2.29843
„ oxalic acid	2.29852
„ formate of soda	2.29854
Mean	2.29850

* A Paper read before the Royal Society, December 9th, 1897.

This corresponds to the number 2.62704 for oxygen (*loc. cit.*, p. 144), and is subject to a correction (additive) of 0.00056 for the diminution of the external volume of the globe when exhausted.

The ratio of the densities of carbonic oxide and oxygen is thus 2.29906 : 2.62760; so that if the density of oxygen be taken as 32, that of carbonic oxide will be 27.9989. If, as some preliminary experiments by Dr. Scott (*Camb. Phil. Proc.*, ix., p. 144, 1896) indicate, equal volumes may be taken as accurately representative of CO and of O₂, the atomic weight of carbon will be 11.9989 on the scale of oxygen = 16.

The very close agreement between the weights of carbonic oxide prepared in three different ways is some guarantee against the presence of an impurity of widely differing density. On the other hand, some careful experiments led Mr. T. W. Richards (*Amer. Acad. Proc.*, xviii., p. 279, 1891) to the conclusion that carbonic oxide is liable to contain considerable quantities of hydrogen or of hydrocarbons. From 5½ litres of carbonic oxide passed over hot cupric oxide he collected no less than 25 m.grms. of water, and the evidence appeared to prove that the hydrogen was really derived from the carbonic oxide. Such a proportion of hydrogen would entail a deficiency in the weight of the globe of about 11 m.grms., and seems improbable in view of the good agreement of the numbers recorded. The presence of so much hydrogen in carbonic oxide is also difficult to reconcile with the well-known experiments of Professor Dixon, who found that prolonged treatment with phosphoric anhydride was required in order to render the mixture of carbonic oxide and oxygen inexplosive. In the presence of relatively large quantities of free hydrogen (or hydrocarbons) why should traces of water vapour be so important?

In an experiment by Dr. Scott (*Chem. Soc. Trans.*, 1897, p. 564) 4 litres of carbon monoxide gave only 1.3 m.grms. to the drying tube after oxidation.

I have myself made several trials of the same sort with gas prepared from formate of soda exactly as for weighing. The results were not so concordant as I had hoped,* but the amount of water collected was even less than that given by Dr. Scott. Indeed I do not regard as proved the presence of hydrogen at all in the gas that I have employed.†

Carbonic Anhydride.

This gas was prepared from hydrochloric acid and marble, and, after passing a bubbler charged with a solution of carbonate of soda, was dried by phosphoric anhydride. Previous to use, the acid was caused to boil for some time by the passage of hydrochloric acid vapour from a flask containing another charge of the acid. In a second set of experiments the marble was replaced by a solution of carbonate of soda. There is no appreciable difference between the results obtained in the two ways; and the mean, corrected for the errors of weights and for the shrinkage of the globe when exhausted, is 3.6349, corresponding to 2.6276 for oxygen. The temperature at which the globe was charged was 0° C., and the actual pressure that of the manometric gauge at about 20°, reduction being made to 15° by the use of Boyle's law. From the former paper it appears that the actual height of the mercury column at 15° is 762.511 m.m.

Nitrous Oxide.

In preliminary experiments the gas was prepared in the laboratory, at as low a temperature as possible, from nitrate of ammonia, or was drawn from the iron bottles in which it is commercially supplied. The purification was by passage over potash and phosphoric anhydride. Unless

* One obstacle was the difficulty of re-oxidising the copper reduced by carbonic oxide. I have never encountered this difficulty after reduction by hydrogen.

† In Mr. Richards's work the gas, in an imperfectly dried condition, was treated with hot platinum-black. Is it possible that the hydrogen was introduced at this stage?

special precautions are taken the gas so obtained is 10 or more m.grms. too light, presumably from admixture with nitrogen. In the case of the commercial supply a better result is obtained by placing the bottles in an inverted position so as to draw from the liquid rather than from the gaseous portion.

Higher and more consistent results were arrived at from gas which had been specially treated. In consequence of the high relative solubility of nitrous oxide in water, the gas held in solution after prolonged agitation of the liquid with impure gas from any supply will contain a much diminished proportion of nitrogen. To carry out this method on the scale required, a large (11-litre) flask was mounted on an apparatus in connection with the lathe so that it could be vigorously shaken. After the dissolved air had been sufficiently expelled by preliminary passage of N_2O , the water was cooled to near $0^\circ C.$, and violently shaken for a considerable time while the gas was passing in large excess. The nitrous oxide thus purified was expelled from solution by heat, and was used to fill the globe in the usual manner.

For comparison with the results so obtained, gas purified in another manner was also examined. A small iron bottle, fully charged with the commercial material, was cooled in salt and ice, and allowed somewhat suddenly to blow off half its contents. The residue drawn from the bottle in one or other position was employed for the weighings.

Nitrous Oxide (1896).

Aug. 15	Expelled from water	3'6359
" 17	"	3'6354
" 19	From residue after blow off,	
	valve downwards	3'6364
" 21	Ditto " upwards	3'6358
" 22	Ditto " downwards	3'6360
Mean		3'6359

The mean value may be taken to represent the corrected weight of the gas which fills the globe at $0^\circ C.$ and at the pressure of the gauge (at 15°), corresponding to 2.6276 for oxygen.

One of the objects which I had in view in determining the density of nitrous oxide was to obtain, if it were possible, evidence as to the atomic weight of nitrogen. It may be remembered that observations upon the density of pure nitrogen, as distinguished from the atmospheric mixture containing argon which, until recently, had been confounded with pure nitrogen (Rayleigh and Ramsay, *Phil. Trans.*, vol. clxxxvi., p. 190, 1895), led to the conclusion that the densities of oxygen and nitrogen were as 16:14.003, thus suggesting that the atomic weight of nitrogen might really be 14 in place of 14.05, as generally received. The chemical evidence upon which the latter number rests is very indirect, and it appears that a direct comparison of the weight of nitrous oxide and of its contained nitrogen might be of value. A suitable vessel would be filled, under known conditions, with the nitrous oxide, which would then be submitted to the action of a spiral of copper or iron wire rendered incandescent by an electric current. When all the oxygen was removed, the residual nitrogen would be measured, from which the ratio of equivalents could readily be deduced. The fact that the residual nitrogen would possess nearly the same volume as the nitrous oxide from which it was derived would present certain experimental advantages. If indeed the atomic weights were really as 14:16, the ratio (x) of volumes, after and before operations, would be given by—

$$\frac{2.2996 \times x}{3.6359 - 2.2996 \times x} = \frac{14}{8},$$

whence—

$$x = \frac{7 \times 3.6359}{11 \times 2.2996} = 1.0061,$$

3.6359 and 2.2996 being the relative weights of nitrous oxide and of nitrogen which (at $0^\circ C.$ and at the pressure

of the gauge) occupy the same volume. The integral numbers for the atomic weights would thus correspond to an expansion, after chemical reduction, of about one-half per cent.

But in practical operation the method lost most of its apparent simplicity. It was found that copper became unmanageable at a temperature sufficiently high for the purpose, and recourse was had to iron. Coils of iron suitably prepared and supported could be adequately heated by the current from a dynamo without twisting hopelessly out of shape; but the use of iron leads to fresh difficulties. The emission of carbonic oxide from the iron heated in vacuum continues for a very long time, and the attempt to get rid of this gas by preliminary treatment had to be abandoned. By final addition of a small quantity of oxygen (obtained by heating some permanganate of potash sealed up in one of the leading tubes) the CO could be oxidised to CO_2 , and thus, along with any H_2O , be absorbed by a lump of potash placed beforehand in the working vessel. To get rid of superfluous oxygen, a coil of incandescent copper had then to be invoked, and thus the apparatus became rather complicated.

It is believed that the difficulties thus far mentioned were overcome, but nevertheless a satisfactory concordance in the final numbers was not attained. In the present position of the question no results are of value which do not discriminate with certainty between 14.05 and 14.00. The obstacle appeared to lie in a tendency of the nitrogen to pass to higher degrees of oxidation. On more than one occasion mercury (which formed the movable boundary of an overflow chamber) was observed to be attacked. Under these circumstances I do not think it worth while to enter into further detail regarding the experiments in question.

The following summary gives the densities of the various gases relatively to air, all obtained by the same apparatus (*Roy. Soc. Proc.*, vol. liii., p. 148, 1893; vol. lv., p. 340, 1894; *Phil. Trans.*, vol. clxxxvi., p. 189, 1895; *Roy. Soc. Proc.*, vol. lix., p. 201, 1896). The last figure is of little significance:—

Air free from H_2O and CO_2	1.00000
Oxygen	1.10535
Nitrogen and argon (atmospheric) ..	0.97209
Nitrogen	0.96737
Argon	1.37752
Carbonic oxide	0.96716
Carbonic anhydride	1.52909
Nitrous oxide	1.52951

The value obtained for hydrogen upon the same scale was 0.06960; but the researches of M. Leduc and of Professor Morley appear to show that this number is a little too high.

ELECTRIC CONDUCTIVITY OF NITRIC ACID.*

By V. H. VELEY, M.A., F.R.S., and
J. J. MANLEY, Daubeny Curator of the Magdalen College
Laboratory, Oxford.

IN this paper an account is given of determinations of the electric conductivity of nitric acid of percentage concentrations varying from 1.3 to 99.97, purified, so far as possible, from reduction products of the acid, as also from sulphuric and the halogen acids, with which it is likely to be contaminated from its process of manufacture. In the preliminary experiments it was observed that the results might be vitiated by (1) a trace of nitrous acid either directly acid or produced by decomposition due to exposure to sunlight, and (2) imperfect insulation of the

* Abstract of a Paper read before the Royal Society, December 9th, 1897.

electrolytic cell caused by metallic clamps, a point which seems to have been neglected by previous observers.

The methods adopted for the purification of the water and nitric acid, as also for the detection and estimation of the impurities, are described in full. The greatest quantity of nitrous acid, sulphuric acid, and the halogen acids found in any sample used were 0.75, 4.3, and 3.8 parts per million respectively.

The thermometers, resistance coils, and other instruments used, were compared with certain standards and corrected accordingly; the burettes and electrolytic cells were calibrated by one or more methods, and the mean of the values accepted.

The method adopted for the determinations was in outline that originally described by Kohlrausch, but modified so as to overcome certain difficulties experienced. A particular form of bridge was constructed, in which the wire was an air line, and a special form of slider adopted to tap without sagging the wire, so arranged that it could be moved by the observer from the extremity of the bridge, and thus all thermo-currents due to his proximity were avoided.

A rapidly revolving commutator was substituted for the usual induction coil, as the latter was found to be unsatisfactory owing to the susceptibility of nitric acid to polarisation.

Various forms of electrolytic cells were used according to the concentration of the acid and the temperature of the observations; these were provided with movable electrodes, so as to throw into circuit different lengths of acid.

A special form of apparatus was devised to prepare nitric acid of 99.88 per cent, and another form to obtain acid of 99.97 per cent from the latter. As a considerable quantity of this practically anhydrous acid was obtained, its chemical and certain physical properties were examined. It has no action on (1) copper, (2) silver, (3) cadmium, and (4) mercury, all of high degree of purity, and (5) commercial magnesium, at ordinary temperatures; purified iron and commercial granulated tin were unaffected by the acid, even when boiling. Purified zinc was slightly acted upon, but sodium immediately caught fire. The acid has no action whatever on calcium carbonate at ordinary temperatures or the boiling-point. Flowers of sulphur and iron pyrites dissolve quickly and completely in the gently warmed acid. The following results were obtained for the density of the 99.97 per cent acid, corrected for weighings *in vacuo* :—

Density $4/4 = 1.54212$; $14/2/4 = 1.52234$; $24/2/4 = 1.50394$, the mean values of two concordant observations.

As a further check upon the measurements obtained by the Kohlrausch method, certain other measurements were made by Carey Foster's method for the comparison of resistances, and the results obtained were found to be concordant within the limits of experimental error. In a series of tables the values are given for thirty-two samples of acid of the specific resistance in true ohms at temperatures of 0°, 15°, and 30°, the temperature coefficients α_{10° and β_{10° deduced from the equation—

$$R_t = R_0(1 + \alpha t - \beta t^2),$$

as also for $K_0 \times 10^9$, $K_{15} \times 10^9$, and $K_{30} \times 10^9$ (the conductivity of mercury at 0 being taken as unity, and its specific resistance as 94.07 microhms per 1 c.c.).

It is shown that the specific resistance decreases for percentage concentrations from 1.30 to 30, at first more, then less rapidly (thus confirming the previous observations of Kohlrausch); from this point the resistance increases slowly up to 76 per cent, thence more rapidly until a maximum is reached at 96.12 per cent, when a sudden reversal takes place.

Further, whereas nitric acid behaves as other electrolytes in possessing a positive temperature coefficient of conductivity for percentage concentrations from 1.3 to 96.12, yet from this point up to 99.97 per cent it behaves

as a metallic conductor in possessing a negative temperature coefficient.

Similar phenomena have been observed by Arrhenius in the cases of moderately dilute solutions of hypophosphorous and phosphoric acids, and explained by him by means of the ionic dissociation hypothesis. It is pointed out that nitric acid of 96 to 99.97 per cent would, *ex hypothesi*, contain few, if any, free ions, and therefore the theory would lead to a totally opposite conclusion.

The results of the experiments are also discussed in relation to the hydrate theory of solution, and the illustrative curves in which the percentages of acid are taken as abscissæ, and the resistances or conductivities in mercury units, show points of discontinuity markedly at percentages corresponding approximately to the composition required for the hydrates $\text{HNO}_3, 2\text{H}_2\text{O}$, $\text{HNO}_3, \text{H}_2\text{O}$, $2\text{HNO}_3, \text{H}_2\text{O}$ ($=\text{H}_4\text{N}_2\text{O}_7$), and less markedly for the hydrate $\text{HNO}_3, 10\text{H}_2\text{O}$. Further, if the values of $\alpha \times 10^4$ and $\beta \times 10^4$ are referred to molecular proportions of water, the minima values of the former and the maxima of the latter occur in the cases of 3.07, 1.84, 0.99, and 0.55 molecular proportions or very approximately—

$\text{HNO}_3, 3\text{H}_2\text{O}$, $\text{HNO}_3, 2\text{H}_2\text{O}$, $\text{HNO}_3, \text{H}_2\text{O}$, and $2\text{HNO}_3, \text{H}_2\text{O}$.

Further evidence is thus added by an independent method to that already accumulated as to the existence of definite combination of nitric acid with water. Finally, it is pointed out that if a curve is plotted out in which the molecular proportions of water are taken as abscissæ, and the values for $\alpha \times 10^4$ as ordinates, there are ascending and descending branches, meeting at the points corresponding to the formation of the respective hydrates; the phenomena are compared with those observed by Bakhuis-Roozeboom for the solubility curves of hydrates of ferric chloride and by Le Chatelier, as also by Heycock and Neville for the freezing-point of alloys.

ON THE OCCLUSION OF HYDROGEN AND OXYGEN BY PALLADIUM.*

By LUDWIG MOND, Ph.D., F.R.S.,
WILLIAM RAMSAY, Ph.D., LL.D., Sc.D., F.R.S., and
JOHN SHIELDS, D.Sc., Ph.D.

DURING their investigations on the nature of the occlusion of gases by finely divided metals, and in particular on the occlusion of hydrogen and oxygen by platinum black, the authors have had occasion to examine the behaviour of palladium to these gases.

The palladium was employed in three states of aggregation, viz., in the form of (a) black, (b) sponge, and (c) foil. Palladium black, prepared in the same way as platinum black, contains 1.65 per cent of oxygen, or, taking the density of palladium black as 12.0, 138 volumes of oxygen. It differs from platinum black, however, inasmuch as the oxygen cannot be removed *in vacuo* at a dull red heat, and consequently had to be determined in the ignited substance by passing hydrogen over it and weighing the water produced. Palladium black dried at 100° contains 0.72 per cent of water, and hence, on the assumption that the oxygen exists as PdO, we have for the analysis of palladium black :—

Pd	86.59 per cent.	
PdO	12.69 ..	= 1.65 per cent O ₂ .
H ₂ O	0.72 ..	

On heating in an atmosphere of oxygen, palladium black goes on absorbing oxygen at least up to a red heat, with the formation of a brownish black substance, which does not again lose its oxygen at a dull red heat *in vacuo*.

* Abstract of a Paper read before the Royal Society, December 16th, 1897.

The amount of oxygen absorbed (nearly 1000 volumes) was about one and a half times as much as corresponds with the formula Pd_2O ; and if the ignition had been sufficiently prolonged, the whole of the palladium would probably have been converted into the oxide PdO .

Palladium black, when exposed to hydrogen gas, absorbed over 1100 volumes, but of this only 873 volumes were really occluded, the remainder having formed water with 139 volumes of oxygen originally contained in the black, which is in good agreement with the direct gravimetric estimation.

Of the hydrogen occluded, about 92 per cent was pumped off slowly at the ordinary temperature, and almost the whole of the remainder at 444° . Increase of pressure of the hydrogen from one atmosphere up to 4.6 atmospheres had no influence on the quantity occluded at the ordinary temperature.

The pure palladium sponge remaining in the experimental tube after the above experiment was over occluded 852 volumes of hydrogen, and about 98 per cent of this was extracted *in vacuo* at the ordinary temperature.

New palladium foil behaved in a very peculiar fashion. At first it scarcely occluded any hydrogen even after ignition in the gas and subsequently cooling down. It was therefore charged and discharged several times electrolytically with hydrogen, but still it persistently refused to occlude any appreciable quantity when replaced in an atmosphere of hydrogen.

After powerful ignition in the blowpipe flame, when it was probably oxidised and then again reduced at a still higher temperature, it was introduced once more into the experimental tube. It immediately occluded a considerable quantity of hydrogen, and by maintaining the temperature between 100° and 130° a large additional quantity was slowly absorbed. On cooling down to the ordinary temperature, hydrogen was again occluded, and it was finally found to have taken up 846 volumes, *i.e.*, approximately the same quantity as the black or sponge.

The hydrogen occluded by palladium foil is given off again very slowly at the ordinary temperature *in vacuo*, but rapidly and almost completely at 100° .

The paper contains some attempts to explain the extraordinary behaviour of palladium foil.

The heat evolved on the occlusion of hydrogen by palladium black was measured in an ice calorimeter (temperature of the room 20 — 24°) in nearly the same way as the corresponding heat of occlusion of hydrogen by platinum black, thereby avoiding errors due to the pre-existence of oxygen in the substance.

Favre's statement that the heat of occlusion remains constant for the different fractions of hydrogen occluded was confirmed, and it was found that $+46.4$ K (4640 g. cal.) were evolved per grm. of hydrogen occluded.

The authors consider that this number may be taken as correct within 1 per cent, and compare it with the different values found by Favre and those calculated by Moutier and Dewar.

If the external work done by the atmosphere be eliminated, the heat evolved per grm. of hydrogen occluded becomes $+43.7$ K.

The heat evolved per grm. of oxygen absorbed was also determined in an indirect manner, and found to be $+11.2$ K (1120 g. cal.).

This number, referred to 16 grms. of oxygen, lies intermediate between the values given by Thomsen for the heat of formation of palladious and palladic hydroxides, and may be consistent, considering the accuracy of such measurements, with the formation of either of these hydroxides or with a mixture of both. In any case it is of the same order of magnitude, and taken in conjunction with the behaviour of palladium black when heated in an atmosphere of oxygen, is undoubtedly in harmony with the view that the absorption of oxygen by palladium black (and probably also by platinum black) is a true phenomenon of oxidation.

The authors have also investigated the atomic ratio—

palladium: hydrogen for fully-charged palladium black, sponge, and foil, and give in tabular form the corresponding ratios deduced from experiments by Graham and Dewar in which wire and block palladium were charged with hydrogen electrolytically. They have arrived at the conclusion that no matter whether the palladium exists as black, sponge, foil, wire, or compact metal, or whether it is charged by direct exposure to hydrogen gas (the proper conditions being observed), or charged electrolytically, the amount of hydrogen occluded in each case is approximately the same, the atomic ratio varying between 1.37 and 1.47.

Hoitsema has shown that Troost and Hautefeuille's deduction that a compound exists having the formula Pd_2H is not warranted. The constancy of the heat of occlusion over the whole range of absorption is also opposed to the view that such a compound is formed.

The composition of fully charged palladium hydrogen corresponds closely with the formula Pd_3H_2 first suggested by Dewar. The principal and almost only evidence, up to the present, in favour of the formation of such a definite chemical compound is to be found in the approximation of the above atomic ratios to the theoretical value 1.5, required by the formula Pd_3H_2 . Although Hoitsema's arguments may be equally well directed against the existence of this compound, the authors consider that additional and independent evidence is desirable, and hope to be able to provide it.

It is also shown that the heats of occlusion of hydrogen in platinum and palladium black are not in favour of the view which has sometimes been put forward that the heat of occlusion of a gas represents the heat of condensation or liquefaction of the gas in the capillary pores of the absorbing substance or the heat of solidification or fusion.

"ON FEHLING'S SOLUTION."

By OTTO ROSENHEIM, Ph.D., F.C.S., and
PHILIP SCHIDROWITZ, Ph.D., F.C.S.

THE *Berichte der Deutschen Chem. Gesellschaft* (vol. xxx., p. 2431) contains a paper by M. Z. Jovitschitsch under the above heading, in which the author asserts that, according to an observation made by Professor Siegfried, mineral acids (hydrochloric, nitric, and sulphuric) possess the property of reducing Fehling's solution—more especially when the latter is still alkaline. Granted that the author's observations are correct, it follows that the reduction is due to the alkali salts of the acids mentioned. As salts of the alkalis are almost invariably present in liquids in which matter capable of oxidation is to be tested for or determined, the experimental confirmation of Jovitschitsch's statements seemed to us to be highly desirable, especially as no analytical data were recorded in his paper.

Viewed theoretically, there is absolutely no reason why salts of the alkalis should influence the reduction of Fehling's solution; and, as might have been expected, both qualitative and quantitative experiments made by us yielded negative results.* It must be assumed that Jovitschitsch's paper is based on an error of observation, and as T. E. Gerock (*Ber.*, xxx., 2865) has meanwhile come to the same conclusion, we refrain from discussing the subject more fully, and merely record the data of some quantitative experiments which are directly to the point. The method adopted was similar to that usually employed in the gravimetric estimation of sugars. Fifty

* Considering that Fehling's solution (in various modifications) has now been in use for close on fifty years, it is indeed remarkable that the phenomena described in the paper quoted have not been observed before. On the contrary, Boivin and Loiseau (*Ber.*, vii., 1790) assert that the presence of small quantities of salts, such as CaCl_2 , BaCl_2 , NH_4Cl , NaCl , &c., prevents the spontaneous reduction that takes place on boiling the solution with distilled water.

c.c. of Fehling's solution (stored in two solutions and mixed when required), diluted with 25 c.c. of distilled water, were heated to boiling, 25 c.c. of a 25 per cent solution of the salt in question run in from a pipette, and after ebullition had re-commenced, the liquid was kept boiling in one series for two, in the other for four minutes. The perfectly clear solution was filtered hot through a Soxhlet's asbestos tube under reduced pressure, the tube dried, heated in hydrogen in the usual manner, and finally weighed. As will be seen from the figures below, the results were practically identical with those obtained by the boiling of Fehling's solution alone.

	Boiled for two minutes. Grm. Cu.	Boiled for four minutes. Grm. Cu.
Fehling's solution alone* ..	0'0028	0'0040
25 per cent potassium chloride ..	0'0023	0'0040
25 per cent potassium nitrate ..	0'0036	0'0044
Potassium sulphate (saturated at 18° C.)	0'0020	0'0040

ON THE NON-EXISTENCE OF AN INTERMEDIATE IODIDE OF MERCURY.

By MAURICE FRANCOIS.

BEFORE 1827 only two iodides of mercury were accepted—the yellowish green mercurous iodide and the red mercuric iodide. At this date Boullay described the intermediate iodide, which he simply called the yellowish iodide, calling the protoxide simply green (*Ann. de Chim. et de Phys.* [2], xxxiv., p. 364, 1827). This new compound of the atomic formula, Hg_4I_6 , has been described as sublimable without decomposition, and as insoluble in boiling alcohol. Boullay has given for its preparation a method, since quoted in all standard chemical works. It consists in adding to a slightly acid solution of mercurous nitrate a solution of iodide of potassium, to which has been added a quantity of free iodine, equal to half the amount it already contains in the state of iodide. Such an operation would undoubtedly give a product of the desired composition, on condition that the precipitation was complete, and that its composition was not changed by the washings. But Boullay recommended stopping the addition of iodide of potassium at the moment when the precipitate, originally yellow, takes a red tint.

I have endeavoured to verify the facts put forward by Boullay, facts which have not up till the present time been contested, and I have arrived at results completely opposed to those of this *savant*. For this research I prepared the compound described by Boullay, by not stopping the addition of the iodised iodide until the precipitate was ended. The precipitate, well washed with water, was collected, analysed, and submitted to further washings with pure ether. I also fractionated the precipitate and analysed the different fractions.

1. I prepared a solution of iodised potassic iodide, containing 166 grms. of iodide of potassium and 63'50 grms. of free iodine per litre. I also made, with freshly prepared and well-dried mercurous nitrate, a solution containing 40 grms. of mercurous nitrate, and 2 c.c. of pure nitric acid per 400 c.c. The iodised solution was put into a burette and allowed to fall slowly, drop by drop, into the solution of mercurous nitrate, which was kept constantly stirred with a glass rod. Each drop made a red splash, which rapidly became yellow.

The colour of the precipitate first formed is of a beautiful yellow; then, as we continue to add the iodised

solution, it passes to a reddish orange, and finally to red. We stop the addition when a drop of the iodised iodide gives no further precipitate in the clear solution, but only a yellow colouration. At this moment we have used 132 c.c. of the iodised solution.

The whole of the precipitate is now red; it is washed with water by decantation, and dried at 60°. It weighs 51 grms. Its composition is as follows:—

Mercury	50'63 per cent.
Iodine	48'50 "

The theoretical composition of the intermediate iodide is:—

Mercury	51'21 per cent.
Iodine	48'79 "

As I stated above, the composition of this precipitate corresponds closely with that of the intermediate iodide.

This precipitate was then washed with cold ether by decantation, in the dark. As I have said elsewhere, this is a method of separating mercurous from mercuric iodide, which is much more exact than that of washing with boiling alcohol, being almost irreproachable. To exhaust 1 grm. of intermediate iodide we use three washings of 50 c.c. each of pure ether, decant each time on a filter, and evaporate the ether under a bell-jar over sulphuric acid, in a small tared crystallising glass. This treatment has removed, from 1 grm. of intermediate iodide, 0'591 grm. of mercuric iodide. The residue, insoluble in ether, is of a very pale yellow colour, and weighs 0'411 grm. Its composition is as follows:—

Mercury	60'85 per cent.
Iodine	38'70 "

The theoretical composition of mercuric iodide is:—

Mercury	61'16 per cent.
Iodine	38'84 "

Thus the so-called intermediate iodide can be split up by ether into mercurous and mercuric iodides.

2. To see better what variations the precipitate undergoes during the gradual addition of the iodised iodide, I carried out a fractional precipitation in the following manner:—

I operated on 40 grms. of mercurous nitrate dissolved in 400 c.c. of water and 20 c.c. of nitric acid. Knowing already that this quantity requires for complete precipitation 132 c.c. of the iodised solution, I used 13'2 c.c. of the latter for each precipitation, so as to obtain ten fractions of the precipitate. The affusion of the iodised iodide is done drop by drop while agitating rapidly.

The first precipitate is thrown on a filter, air-dried with a filter-pump, and the filtrate treated as before for the second precipitate, and so on to the end. Each precipitate is carefully washed with water, and dried at 60°.

Analysis gave the following figures:—

- 1st fraction.—Pure bright yellow colour. Mercury 61'02 per cent. Iodine 38'66 per cent.
- 2nd fraction.—Pure bright yellow colour. Mercury 61'29 per cent. Iodine 38'50 per cent.
- 3rd fraction.—Bright yellow. Mercury 61'19 per cent. Iodine 38'19 per cent.
- 4th fraction.—Bright red. Mercury 44'23 per cent. Iodine 55'32 per cent.
- 5th fraction.—Bright red. Mercury 44'81 per cent. Iodine 54'55 per cent.
- 6th fraction.—Bright red. Mercury 44'95 per cent. Iodine 54'80 per cent.
- 7th fraction.—Bright red. Mercury 45'72 per cent. Iodine 53'65 per cent.
- 8th fraction.—Bright red. Mercury 45'93 per cent. Iodine 54'00 per cent.
- 9th fraction.—Bright red. Mercury 44'51 per cent. Iodine 55'02 per cent.
- 10th fraction.—Bright red. Mercury 44'30 per cent. Iodine 55'56 per cent.

* It is a well-known fact that even the most carefully prepared Fehling's solution gives a slight precipitation of cuprous oxide on heating, varying in amount from 2 to 3 mgms. (see, also, Brown, Morris, and Millar, *Journ. Chem. Soc.*, lxi., 96).

The theoretical composition of mercurous iodide is:—

Mercury	61·16 per cent.
Iodine	38·84 „

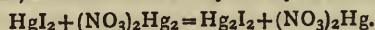
and that of mercuric iodide:—

Mercury	44·05 per cent.
Iodine	55·55 „

As is easily seen, the first three fractionations are of pure mercurous iodide, the others of fairly pure mercuric iodide. It is worthy of remark that the mercurous iodide obtained in this manner is of a very pure yellow colour.

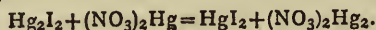
This reaction, and the successive appearance of the mercurous and mercuric iodides, can be explained by the two following experiments.

When we put about 1 grm. of mercuric iodide, recently precipitated and washed but not dried,—that is to say, in the finest possible state of division,—into 100 c.c. of our mercurous nitrate solution at 40 grms. per 400 c.c., we notice, after about fifteen minutes, that the red mercuric iodide changes to a pure yellow colour, being transformed into mercurous iodide. There is an increase in weight of the iodide, and the reaction may be expressed thus:—



This is the transformation of the red splash into yellow, already mentioned above.

When, on the contrary, we put 1 grm. of mercurous iodide into 100 c.c. of a solution of mercuric nitrate containing 40 grms. of nitrate per 400 c.c., we notice that, after about fifteen minutes, the yellow mercurous iodide changes into the red mercuric iodide, according to the equation—



There are thus two limiting contrary reactions.

But when we pour the iodised solution of potassic iodide into mercuric nitrate, the free iodine produces mercuric nitrate, since the mercury of the mercurous nitrate cannot be precipitated except by producing mercuric nitrate or nascent oxygen, which comes to the same thing.

We have thus a liquid in which the proportion of mercurous nitrate goes on diminishing, while the proportion of mercuric nitrate goes on increasing: this sufficiently explains why the nature of the precipitate changes at a given moment, and why the precipitate is at first mercurous iodide and afterwards mercuric iodide. This oxidising action of free iodine seems to have escaped Boullay.

It seems extraordinary that a chemist of such repute as Boullay should have been mistaken on this point. Being fully persuaded that mercurous iodide is green, and the greener it is the purer it is, he could not admit that a distinctly yellow substance was mercurous iodide. Further, he was at this time much preoccupied in proving the existence of double iodides, in which one of the iodides plays the part of an acid, and the other the part of a base; the existence of an intermediate iodide fitted in with his theory, for he regarded it as a combination of mercurous and mercuric iodides. One feels convinced after reading Boullay's paper that he established the existence of this body by relying on his theoretical notions, and on the colour more than on analysis, for he only gives one estimation of mercury, by means of metallic iron, the iodine being estimated by difference. Moreover, the substance analysed by him had not been previously purified.

Conclusions.—In attempting to prepare the intermediate iodide by Boullay's method (1), we obtain a mixture of mercurous and mercuric iodides, separated by ether; (2), the first portions of the precipitate are pure mercurous iodide, to which succeeds pure mercuric iodide.

The intermediate iodide of mercury is therefore not a chemical compound, but a mixture, the more yellow and

rich in mercurous iodide in proportion as the precipitation has been pushed less far.—*Journ. de Pharm. et de Chim.*, Series 6, vol. vi., No. 10, 1897.

ON CERIUM.

By O. BOUDOUARD.

I HAVE the honour of submitting to the Academy the results of researches on the salts of cerium. Continuing the work which I have undertaken with my regretted master, Paul Schützenberger, I have chiefly studied the cerium acetate and sulphate.

Cerium Acetate.

174 grms. cerium sulphate, free from thorium, are dissolved in water and treated with the corresponding quantity of lead acetate, to obtain cerium acetate. The excess of lead is removed by means of a current of hydrogen sulphide. After filtering off the lead sulphide, the ceric solution is allowed to settle in the cold, and in a very short time deposits an abundant white precipitate.

A portion of this acetate was converted into sulphate, and the sulphate was analysed by ignition. I have thus found the atomic weight of the corresponding metal—

$$\text{Ce} = 137\cdot85.$$

Having made fractionated crystallisations of this sulphate, I obtained—

First crystallisation	Ce=140·7
Mother-liquor	Ce=138·5

In all the crystallisations which have been effected the mother-liquors were always precipitated by alcohol. The sulphate thus obtained, after desiccation, is dissolved in cold water and again crystallised; this precaution is taken in order to obtain salts absolutely neutral.

The clear solution obtained from the filtration of the basic acetate was concentrated in the water-bath. The first deposit formed gave on analysis:—

First deposit .. {	Crystallisation (a) ..	Ce=137·35
	Mother-liquors (a) ..	Ce=135·1

Proceeding then with a series of fractionated crystallisations, I obtained the following results:—

Second deposit {	Crystals (b)	Ce=136·5
	Mother-liquors (b) ..	Ce=137·4
Mother-liquors. {	Crystals (c)	Ce=139·1
	Mother-liquors (c) ..	Ce=136·05

Using Peroxide of Hydrogen.—If to a solution of acetate of cerium we add an excess of peroxide of hydrogen, a yellow precipitate is formed; this precipitation is facilitated by heat, which, however, should not be kept up for too long a time; and, further, the precipitation is only partial.

In an experiment I made, I obtained 6 grms. of oxide, which was transformed into sulphate and submitted to fractional crystallisations:—

First crystallisation	Ce=137·15
Mother-liquor	Ce=137·6

The oxides were mixed afresh, transformed into sulphates, and these submitted to another fractional crystallisation, which gave—

First crystallisation	Ce=137·15
Second „	Ce=137·35
Third „	Ce=137·6

Oxalic acid was added to the part not precipitated by peroxide of hydrogen. The oxalate of cerium was calcined and the oxide transformed into sulphate. Fractional crystallisations gave—

First crystallisation	Ce=137·85
Second „	Ce=139·9
Third „	Ce=138·85

Sulphate of Cerium.

A solution of cerous sulphate, to which had been added 20 grms. of sulphate of potassium, produced a double sulphate, which I will call S.D. No. 1. The clear liquid was re-precipitated by means of an equal quantity of sulphate of potassium; this gave me the precipitate S.D. No. 2. Continuing in this manner, I obtained S.D. No. 3 and S.D. No. 4; the wash waters from this last precipitate contained nothing further. Each of these double sulphates was decomposed by caustic soda; the hydrate obtained was washed with warm water, and finally dissolved in nitric acid and precipitated by oxalic acid. This oxalate was calcined, and the oxide transformed into sulphate. I thus made a series of crystallisations with the following results:—

S.D. No. 1.—First crystallisation..	..	Ce=138.75
Second	..	Ce=137.3
Third	..	Ce=133.0
S.D. No. 2.—First	..	Ce=138.5
Second	..	Ce=136.95
Third	..	Ce=137.9
Fourth	..	Ce=137.7
S.D. Nos. 3 and 4.—First crystallisation	..	Ce=138.25
Second	..	Ce=136.25

All these results, whether obtained with acetate or sulphate of cerium, show that, conformably with the indications already given by Schützenberger (*Comptes Rendus*, cxx., p. 962), oxide of cerium is accompanied by small quantities of another earth with a lower atomic weight. This earth should be susceptible of giving a binoxide by oxidation, and its sulphate should give double sulphates insoluble in the alkaline sulphates.

Further, peroxide of hydrogen separates an oxide, of which the atomic weight of the corresponding metal varies from 137.15 to 137.6; while the part not precipitated gives atomic weights varying from 137.85 to 139.9—variations of the same character as those obtained with the double sulphates (from 133.0 to 138.75) and with the acetate (from 135.1 to 140.7).—*Comptes Rendus*, cxxv., No. 20.

ON THE CHLORONITRIDES OF PHOSPHORUS.*

By H. N. STOKES.

(Concluded from p. 311).

Heptaphosphonitric Chloride, $P_7N_7Cl_{14}$.—After many distillations, this gave—

	Calculated for $P_7N_7Cl_{14}$.	Found.
P	26.75	26.57
N	12.11	12.12
Cl	61.14	61.28

Ratio, P : N : Cl = 1 : 1.01 : 2.02.

Molecular Weight. Solvent: Benzene.

Grms. solvent.	Grms. substance.	Elevation.	Molecular weight.	Percentage variation from theoretical.
47.09	3.5621	0.250°	808	-0.5
"	7.3979	0.523°	802	-1.2

Calculated for $P_7N_7Cl_{14}$, 811.7.

Heptaphosphonitric chloride is a nearly colourless rather viscous liquid, which does not solidify at -18° , and which boils at $289-294^\circ$ (corr.) at 13 m.m., undergoing some polymerisation. It is readily miscible with benzene, gasoline, and ether, and towards water shows the stability manifested by the preceding chloronitrides.

Residual Oily Phosphonitric Chlorides.†—The residue

which did not distil at 13 m.m. when the temperature of the bath was 370° , was boiled with water to remove the solid polyphosphonitric chloride, filtered, and carefully dried *in vacuo* over sulphuric acid. It is a thick liquid which gave on analysis:—

	Calculated for $(PNCl_2)_x$.	Found.
P	26.75	26.79
N	12.11	12.39
Cl	61.14	62.00

Ratio, P : N : Cl = 1 : 1.02 : 2.02.

Although the oil is doubtless a mixture, the above figures show that the constituents are members of the series $(PNCl_2)_n$. A determination of the mean molecular weight was made, with the following results:—

Grms. solvent.	Grms. substance.	Elevation.	Molecular weight.
46.72	4.130	0.178°	1326
46.72	7.853	0.348°	1290

Mean 1308. This does not lie far from that required by the formula $P_{11}N_{11}Cl_{22}$ (calculated, 1276). This result is interesting in as far as it shows that a phosphonitric chloride of this molecular weight may exist, that it is stable and miscible with benzene, gasoline, and ether, and that the molecular weight of the solid polymer described below, which is insoluble, is probably very much higher. The oil has a reddish brown colour, due to dissolved impurities, which are destroyed by heating with strong nitric acid. It cannot be distilled even at 13 m.m., as it polymerises almost instantly. In its behaviour towards water it resembles the preceding members of the series.

Polyphosphonitric Chloride, $(PNCl_2)_x$. This remarkable body, frequently alluded to above, is formed when any of the lower members are heated; slowly at 250° , and very rapidly at 350° . As the change is reversible, complete transformation cannot be effected, but reaches perhaps 90 per cent, the remainder consisting not only of the original phosphonitric chloride, but of others. These can be extracted by anhydrous benzene. The sample, the analysis of which is given, was prepared by heating pure triphosphonitric chloride in a sealed tube at $350-360^\circ$. The transparent elastic product was repeatedly extracted with benzene, dried over sodium, and the absorbed benzene removed in a vacuum with constant exhaustion over paraffin, and finally by heating *in vacuo* at 110° . Analysis gave—

	Calculated for $(PNCl_2)_x$.	Found.
P	26.75	26.78
N	12.11	12.27
Cl	61.14	60.45

Ratio, P : N : Cl = 1 : 1.01 : 1.98.

Polyphosphonitric chloride, when perfectly pure, is colourless and transparent, but is generally somewhat discoloured by traces of organic matter. Its most striking property is its elasticity. It may be drawn out like rubber, and shows even a greater tendency to rebound from hard surfaces. It is readily cut with the shears. It is insoluble in all neutral solvents, but absorbs benzene, swelling to many times its original volume and forming a jelly of but little coherence; on evaporating the benzene it returns to its original condition; ether is absorbed, but less readily, and other chloronitrides are taken up in a similar manner. Hot water slowly dissolves it with decomposition; in warm dilute ammonia it swells, gelatinises, and finally dissolves; hot caustic soda does not dissolve it readily, apparently insoluble sodium salts being formed. It begins to depolymerise towards 350° , and this change is rapid just below red heat, and is accompanied by partial fusion, the products being, as described above, a mixture of lower phosphonitric chlorides. These transformations are not modified by heating in an atmosphere of hydro-

* Published by permission of the Director of the United States Geological Survey. From the *American Chemical Journal*, vol. xix., No. 9, November, 1897.

† It is unlikely that these consist entirely of the original depolymerisation products; a portion is doubtless formed by polymerisation during distillation.

chloric acid. When perfectly pure, it leaves no residue whatever on distilling. No difference could be detected in the product formed from different phosphonitrilic chlorides.

Nitrilo-hexaphosphonitrilic Chloride, $P_6N_7Cl_9$.—The separation of this chloronitride, which does not belong to the phosphonitrilic chloride series, is described above. After four crystallisations from benzene, it gave—

		Calculated for $P_6N_7Cl_9$.	Found.
P		30.84	30.74
N		16.29	16.29
Cl		52.87	53.01

A molecular weight determination, kindly made for me by Dr. H. C. Jones, of the Johns Hopkins University, with a limited quantity of material, in benzene solution, gave 667; calculated for $P_6N_7Cl_9$, 603.5. This, with the analysis, suffices to establish the above molecular formula.

Nitrilo-hexaphosphonitrilic chloride strikingly resembles the phosphonitrilic chlorides. It fuses at 237.5° (corr.) and boils at $251-261^\circ$ (corr.) at 13 m.m. without change. The boiling-point coincides closely with that of hexaphosphonitrilic chloride ($261-263^\circ$ corr. at 13 m.m.), hence it is found associated with the latter. Heated in small quantities on foil, it volatilises without residue, but at a higher temperature in a sealed tube it undergoes a change the exact nature of which has not been determined, but which involves the formation of a substance resembling polyphosphonitrilic chloride, which yields lower phosphonitrilic chlorides on distilling. It crystallises in transparent prisms of not more than 1 m.m. in length, apparently rhombic, which are often united to acicular forms. When pulverised it becomes electrified. It dissolves in about 20 parts cold and 5 parts boiling benzene (approximate only), is more soluble in carbon disulphide, but less soluble in gasoline and in ether. Towards water it is nearly as stable as hexaphosphonitrilic chloride, but is slowly attacked when exposed to atmospheric moisture. Hot dilute ammonia dissolves it very slowly, but more rapidly when alcohol is added.

This body is obviously a secondary product of the reaction of phosphorus pentachloride and ammonium chloride, as it is never found when a pure phosphonitrilic chloride is polymerised and depolymerised. It is noteworthy that no indication of other bodies of a similar nature has been observed, although no reason appears why they should not be formed at the same time. Whether it is in reality hexaphosphonitrilic chloride in which three chlorine atoms are replaced by one of nitrogen or not, cannot be decided at present.

Examination of Auriferous Ores.—The gold present in such ores can be rapidly and safely determined by the following method, which is applicable to all kinds of ores:—75 grms. of borax (anhydrous), the same weight of crude tartar, and 50 grms. of red lead are mixed; 250 grms. of the pulverised ore are then added, along with 100 grms. of potash. This mixture is placed in a J crucible, and above it is spread a stratum of 75 grms. soda, and over this again a layer of sodium chloride 1.5 c.m. in thickness; lastly, 50 grms. of red lead. The crucible is placed in a gas-furnace, cautiously heated, at last strongly enough to bring it to the state of quiet flux. The melted mass is poured into a conical form, at the bottom of which it arrives as a lead regulus, which after the treatment is cupelled in a muffle furnace. If the ore contains no silver, a little silver free from gold is added. The globule of silver—possibly auriferous—is treated with nitric acid. The silver dissolves, and the residual gold is weighed. —*Chem. Zeitung* and *Swed. Teknisk. Tid.*

REGULATIONS OF THE

THIRD INTERNATIONAL CONGRESS OF APPLIED CHEMISTRY IN VIENNA, 1898.

1. THE Third International Congress of Applied Chemistry will be held in July, 1898, in Vienna, and will last about five days.

2. The subjects of the Congress are as follows:—

Consultations concerning important questions in all departments of Applied Chemistry, and particularly of those the solution of which is a matter of public interest.

Agreement upon methods to be considered internationally valid for the analysis of such products as are valued upon the basis of their chemical composition.

Agreement upon methods for the use of the different chemical industries, to be considered internationally valid.

Discussion on questions of instruction in Applied Chemistry, and consultations upon general affairs of Chemistry; and commencement of a friendly understanding between the representatives of the different departments of Applied Chemistry at home and abroad.

3. For the settlement of the tasks of the Congress there will be two general meetings and a greater number of special consultations. Besides these there will be excursions for the purpose of visiting scientific institutions and establishments.

4. The special consultations of the Congress will be divided into twelve Sections.

Section I. General Analytical Chemistry and Chemical Instruments. (General methods of analysis; analytical apparatus, measuring instruments, hydrometers, &c.)

Section II. Chemistry of Food, Medical and Pharmaceutical Chemistry. (The chemical and physical analysis of foods, discussion of chemical questions regarding foods in trade which do not come under the head of another section; further questions of medical and pharmaceutical chemistry).

Section III. Agricultural Chemistry. (Agricultural chemistry; experiments and analysis of dairy products).

Section IV. Sugar Industry, Starch and Grape Sugar Manufacture.

1. Brewery and Malt Manufacture.

2. Alcohol and Yeast Industry.

Section V. Zymology.

Section VI. Ceno-Chemistry.

Section VII. Chemical Industry of Inorganic Substances. (Sulphuric acid, soda, and chloride of lime manufacture; alkaline industry; artificial manure production; lime and cement industry; industry of illuminants; glass, china, and earthenware manufacture).

Section VIII. Metallurgy and Industry of Explosives.

Section IX. Chemical Industry of Organic Substances. (Industry of aniline dyes; dyeing and printing; manufacture of pharmaceutical preparations; chemistry of fats, oils, and lubricants; paper and xylonite industry; tannery and glue manufacture).

Section X. Chemistry of the Graphic Industry. (Photochemistry, photographic and chemical printing, colour printing, &c.).

Section XI. Questions of instructions and general affairs of chemists.

Section XII. Electro-chemistry.

Questions which belong to several departments at once will be discussed in common meetings of the Sections concerned.

5. Anyone can become member of the Congress who is concerned with the theory or practice of chemistry or practically employed in any department of chemistry, as well as such persons and corporations as are interested in an undertaking in which chemical processes are applied, and also all those persons and societies which take an interest in the progress of Applied Chemistry.

6. Every member has to pay a fee of 10 florins, for

which he receives a members' card, which admits him to the general meetings and special sessions and all other Congress arrangements and entitles him to all publications of the Congress. Members who subscribe at least 100 florins for the benefit of the Congress will be mentioned specially in the Congress publications as Patrons of the Third International Congress for Applied Chemistry, Vienna, 1898.

7. Any surplus of the subscriptions will be devoted to benevolent purposes, and this will be decided at the second general meeting of the Congress.

8. Every member must enter his name at the beginning of the Congress in the lists of those Sections in which he will take part, and at the same time give his address during the Congress.

9. Every member of the Congress has a right to take part in the debates of the special consultations, and has the right of voting in the general meetings and the consultations of those Sections of which he is a member.

10. The languages to be spoken at the Congress are English, French, and German.

11. The business of the Congress will begin at the first general meeting. This will be opened by the President of the organising committee, and the record will be kept by the Council of the organising committee.

This meeting elects the Honorary President and the Honorary Vice-President, and nominates the President, the Vice-Presidents, the General Secretary, and Secretaries of the Congress.

12. The second general meeting will take place at the end of the Congress. This will be opened and presided over by the President of the Congress. The record will be kept by the General Secretary and the Secretaries of the Congress. In this an account will be given of the particulars of the Congress by the General Secretary, and the meeting will decide place and time of the Fourth International Congress of Applied Chemistry.

13. The first sitting of each branch section or sub-section will be opened by the President nominated by the organising committee, and the formation of the council of the special section or sub-section will be introduced by him. For the formation of the latter a President and a corresponding number of Vice-Presidents must be elected for the first sitting, and once for all a chief Secretary and several secondary Secretaries (according to the length of the discussions) for all the sittings of the section or sub-section. At the end of each sitting the President and the Vice-President for the following sitting are to be elected.

The Presidents, Vice-Presidents, and Secretaries elected by the organising committee remain at the same time members of the council of the special section or sub-section during all the sittings of the same.

14. The order of the day and the succession of the subjects of the consultation will be arranged independently by each section and sub-section. Reports which are printed and distributed to the members beforehand are generally not read aloud, and the debate upon these will be commenced at once by the President. Verbal reports should not last longer than twenty minutes. In the debate a speaker may not speak longer than ten minutes, and it is not permitted that he should speak on the same subject more than twice. The voting will be based on the members' lists of the special section or sub-section, and the simple majority decides. In case of equal votes the President gives the casting vote.

15. For the purpose of drawing up the Congress Record those reporters whose reports were not printed, as well as all speakers in the debate, must give a short account in writing of their speech to the first Secretary of the special section or sub-section not later than half an hour after the close of the meeting in question. The first Secretary must draw up the Record by means of the accounts received, and give it up if possible on the same day, together with the list of those present, to the General Secretary of the Congress.

16. The addresses to be given during the Congress, and

the reports to be presented, must be announced, not later than 30th of April, 1898, to the Special President of the section, or direct to the organising committee. Later announcements can only be accepted by the decision of the branch section or sub-section of the Congress concerned.

Lectures or reports which have to be printed must be sent at the latest on the 15th of April, 1898, to the General Secretary of the organising committee or to the President of the section concerned. The reports must not take more than five 8vo pages in print. The organising committee will decide regarding manuscripts of greater length of purpose for printing.

17. All conclusions or resolutions of the Congress will be laid before the respective Governments by the organising committee.

18. After the conclusion of the Congress a printed report will be given, free of charge, to all members.

19. About all other matters concerning the Congress not mentioned in the Congress Regulations the organising committee will give every information.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Extra Meeting, December 15th, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

PROFESSOR F. R. JAPP, LL.D., F.R.S., delivered the
Kekulé Memorial Lecture.

FRIEDRICH AUGUST KEKULÉ was born at Darmstadt on September 7th, 1829. Originally intended for the profession of an architect, he was induced, by hearing Liebig's lectures, to devote himself to chemistry. After studying under Liebig, he spent a year in Paris, where he became intimate with Gerhardt. Later on he resided for a time in London, making the acquaintance of Williamson and Odling. He always acknowledged the influence which these three chemists had exercised on the formation of his opinions. Kekulé's theories are based on Gerhardt's type theory; on Williamson's theory of polyvalent radicles, which, by their power of linking together other radicles, render possible the existence of multiple types; and on Odling's theory of mixed types, which was a deduction from Williamson's theory. Less consciously perhaps his opinions were influenced by E. Frankland's theory of the valency of elementary atoms, and by Kolbe's speculations on the constitution of organic compounds. Kekulé gathered together the various ideas which he found scattered throughout the writings of his predecessors, added to them, and welded the whole into the consistent system which forms our present theory of chemical structure. In 1857, in the course of a memoir on the constitution of fulminic acid, he gave a tabular arrangement of compounds formulated on the type of marsh gas, this being the earliest statement, although put forward only in an imperfect form, of the tetravalency of carbon. In the same year he published an important theoretical paper: "On the so-called Conjugated Compounds and the Theory of Polyatomic Radicles, which contains a complete system of multiple types and mixed types. In 1858 the celebrated paper, "On the Constitution and Metamorphoses of Chemical Compounds, and on the Chemical Nature of Carbon," appeared; it embodies the fully-developed doctrine of the tetravalency of carbon, together with Kekulé's views on the linking of atoms and on the valency of such chains of atoms—the foundation on which our modern system of constitutional formulæ rests. In 1865 Kekulé put forward his well-known benzene theory—the crowning achievement, in his hands, of the doctrine of the linking of atoms, and the

most brilliant piece of scientific prediction to be found in the whole range of organic chemistry. The conception of closed chains, or cycloids, which he thus introduced, has shown itself to be capable of boundless expansion.

Kekulé published the first instalment of his "Lehrbuch der Organischen Chemie" in 1859. The work was never finished, but it was instrumental in widely disseminating Kekulé's views, and exercised enormous influence on the development of the science.

Kekulé obtained the *venia legendi* in Chemistry at the University of Heidelberg in 1856. Two years later he was called, as ordinary professor, to the University of Ghent, where he remained until 1867, when he was appointed to the Professorship of Chemistry in the University of Bonn, a post which he continued to hold until his death on July 13, 1896. During his later years he suffered from bad health.

The characteristic note of Kekulé's great theoretical creation, the chemistry of structure, is the treatment of the problem of isomerism—the problem which first necessitated the use of constitutional formulæ—as one of geometrical symmetry. Kekulé's formulæ, freed from the fetters of the type theory with which he had first encumbered them, were merely more or less symmetrical geometrical figures. In order to predict the number of substitution compounds, it was only necessary to consider the degree of dissymmetry of the parent compound: the less the symmetry, the greater the number of isomeric substitution compounds. The extraordinary fertility of this conception is shown by the development which it has undergone at the hands of van't Hoff, J. Wislicenus, von Baeyer, and others.

The accuracy of Kekulé's predictions has done more to inspire a belief in the utility of legitimate hypotheses in chemistry, and has therefore done more for the deductive side of the science, than that of almost any other investigator. His work stands pre-eminent as an example of the power of ideas. A benzene formula, consisting of a few chemical symbols jotted down on paper and joined together by lines, has supplied work and inspiration for scientific organic chemists during an entire generation, and affords guidance to the most complex industry the world has yet seen.

Dr. HUGO MÜLLER, as probably the oldest personal friend of Kekulé present, moved a cordial vote of thanks to Professor Japp for his eloquent lecture, and added his special appreciation of the admirable and exhaustive manner in which the lecturer had accomplished his task. A considerable effort was needed to realise the condition in which organic chemistry stood fifty years ago, in order to recognise the vast advances which have been made in the interval. It may be truly claimed for Kekulé that he holds a foremost position amongst those reformers who have initiated this progress. It was here, in London, that Kekulé first conceived the ideas which, in their further development, assumed the shape of his "chemistry of carbon" and "benzene theory," and, being in those days in almost daily intercourse with him, he well recollected the eagerness and enthusiasm with which the problems which occupied his mind were discussed.

Soon afterwards, in Heidelberg, and then in Ghent, his affable manner and sociability attracted a number of devoted pupils, who became active fellow workers, and thus his teaching bore fruit in all directions.

Unfortunately, not long after he had been settled in Bonn, his health gradually gave way, and he suffered much from nervous prostration and an irksome degree of deafness, which at times much depressed him. His power of work became greatly impaired, and notwithstanding repeated heroic efforts, even his Handbook had to remain unfinished.

Professor THORPE, in seconding the resolution, also desired to give expression to the sense of obligation which the Society was under to Professor Japp for the thoughtful and eminently impartial address which he had

given. There was, however, one slight but characteristic omission in the lecture. In enumerating Kekulé's students, Dr. Japp had neglected to make any reference to himself. It was, no doubt, that same feeling of piety to which he had borne witness in the course of his lecture on the part of another which induced Dr. Japp to comply so readily as he had done with the request of the Council that he should undertake the weighty and responsible duty of delivering this address. His personal intercourse, as a student, with the master had, we may take it, quickened his appreciation of his work. At the same time, as would be evident, it had in no sense diminished his critical faculty. The audience had recognised that the lecture was a truthful and well-balanced account, written impartially and in the true spirit of history, of the origin and fruit, so far as this had been gathered, of the great chemist's labours.

Some reference had been made to the fact that he (the speaker) had enjoyed the good fortune of also being a student under Kekulé, and of being associated with him, in some small degree, in certain experimental work which he undertook during the first years of his professorship in the magnificent institution which Germany owes to Hofmann. It is a curious coincidence that Hofmann, like Kekulé, might have become an architect if destiny, as in Kekulé's case, had not intended that he should be a chemist. During the late sixties there was no sign of the decay in intellectual vigour which a few years later became so sadly obvious. At that time the great generalisation which we associate with Kekulé's name was still, to some extent, on its trial, and it had to withstand the assaults which were from time to time delivered by keen and active opponents in other schools of chemical thought. It happened that very shortly after the speaker's entrance into Kekulé's laboratory he was called upon to handle the weapons which Kekulé himself placed in his hands in order to defend a small but apparently vulnerable point in the theory. That circumstance proved of incalculable benefit to him, in that it brought him into intimate personal contact with Kekulé, and enabled him to see something of his methods of work, and of the springs of his intellectual activity. Dr. Japp has ably testified to Kekulé's merits as a teacher. Kekulé, indeed, was one of the very best expositors, with the single possible exception of Kirchhoff, to whom it had been the speaker's lot to listen. As a laboratory teacher he was excellent. He was a most severe judge of work, striving to exact the same high manipulative finish, the same neatness and order, which he invariably bestowed on everything he did, and he was absolutely intolerant of anything slovenly or "sloppy." But it was as a lecturer that he was seen at his best. He was singularly luminous as a thinker, a close and accurate reasoner, with a remarkable power of concentrated expression. He was not a rapid speaker, and he never indulged in those rhetorical flights with which Hofmann occasionally was wont to electrify an audience. His language was apt and well chosen, and his delivery easy and natural. His lecture-table was never overburdened with "experiments"; those he showed were strictly proper to the subject in hand. To see him handle the chalk was in itself a liberal education. Although everything appeared to be so easy and natural, an attentive critic could hardly fail to perceive that the lecture had been carefully thought out beforehand, possibly over a longer period of time than it took to utter. Every detail would seem to have been considered, even to the particular places on the blackboard where the formulæ should appear.

During the later period of his life, Kekulé, unfortunately for Science, was comparatively sterile. Those who knew him, however, would be the first to affirm that this seeming apathy sprang from no natural indolence. There is no doubt that he suffered, even in the early period of middle life, from the intense stress and strain of his mental labours prior to the Ghent period. He had too surely exemplified the sad truth of Liebig's

saying, to which Dr. Japp had referred, that he who would become a great chemist must pay for his pre-eminence by the sacrifice of his health. There is reason to know that it was the consciousness of failing power which prevented him from finishing much to which he had put his hand, and that his fastidiousness and his sense of "finish," amounting almost to hypercriticism, restrained him from publishing what he realised fell short of his ideal. What he has left us, however, is an imperishable monument to his genius.

The PRESIDENT said that there was little to add to Professor Japp's exhaustive eulogy of the life and work of Kekulé. His own early relations, however, with the great chemist whose life work the Society was commemorating might have some interest to the members and ought to be told. While a student with Lord Playfair at Edinburgh, in the session 1866-67, he made his first contribution to Science in the shape of a little paper entitled, "On the Oxidation of Phenyl Alcohol and a Mechanical Arrangement adapted to Illustrate Structure in the Non-saturated Hydrocarbons." This note appeared in the *Proceedings of the Royal Society of Edinburgh*, and he was so desirous of becoming known to Kekulé as a student of his theory of the aromatic bodies that a specimen model was sent to Ghent. Lord Playfair addressed a letter to Kekulé stating that he (Professor Dewar) was very anxious to work in his laboratory. The reply was, "Come," and the reception and kindness he received from Kekulé has always had his profound gratitude.

The summer of 1867 was thus spent in the private laboratory of Kekulé. Before leaving Edinburgh, he had been working on the coal tar bases, and a supply was taken with him to Ghent. There he began the study of the oxidation products of picoline, and at the British Association Meeting at Norwich, in 1868, an account of the separation of dicarbopyridinic acid, the analogue of phthalic acid in the benzene series, was given. At the same meeting, he gave a paper on "Kekulé's Model to Illustrate Graphic Formulæ." This is the succinct history of the beginning of the pyridine-benzene analogy. His old friend Koerner had speculated in the same direction, and he (Prof. Dewar) might confess that in his opinion they both had received too much credit for an extension of the benzene theory to pyridine. At a distance of thirty years, to look back and call to mind the presence and personality of the great chemist as he knew him was indeed a pleasure. He was a man of noble mien, handsome, dignified, and yet of a homely and kindly disposition. He was a severe critic, having a haughty contempt for the accidental and *bizarre* in scientific work. His originality and suggestiveness seemed endless, so that he had no need to commit scientific trespass or to follow just in the wake of other people's ideas. Everything that passed through the Kekulé alembic was indeed transmuted into pure gold. His precision of thought and diction rendered his papers profoundly suggestive to other workers. His great work will always live in the history of our Science, and his loving memory will be for ever enshrined in the hearts of his pupils.

CORRESPONDENCE.

THE EXPLOSION AT DARTFORD.

To the Editor of the Chemical News.

SIR,—Having received numerous enquiries in respect to the fatal explosion which occurred at our Dartford works on Wednesday, the 15th inst., and so many erroneous versions having appeared in the public press, we think it well to state the simple facts of the case.

The deceased, Mr. Lewis Jones (qualified chemist), was

mixing a preparation of erythrol tetranitrate—a remedy which is now somewhat extensively prescribed by the medical profession in cardiac affections. The process consisted in diluting erythrol tetranitrate with finely-powdered lactose by gently stirring the substances in a mortar; no pounding or grinding was required. The quantity of erythrol in the possession of the deceased was four ounces. Our process for dealing with this substance was adopted after a series of careful experiments, and it has always been performed by competent chemists who knew the dangers of such nitrous compounds. The process has been carried out by us many times during the past eighteen months without the slightest mishap. It has been the rule of the chief of the department to caution those who handled it; and the deceased received such a warning. The erythrol tetranitrate was kept under lock and key in a dark closet. The cause of the explosion we can only attribute to some extraordinary accident; unfortunately there was no witness to the actual carrying out of the operation by the deceased on this occasion. The force of the explosion was violent, but local.

We need hardly add that we are deeply pained by this sad occurrence, which has resulted in the death of an employé who had gained our high esteem.—We are, &c.,

BURROUGHS, WELLCOME, & Co.

Snow Hill Buildings, London, E.C.,
December 21, 1897.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 20, November 15, 1897.

Reaction of Hydrogen upon Sulphuric Acid.—M. Berthelot.—The author finds that there is an important reaction between hydrogen and sulphuric acid. 0.5 gm. of H_2SO_4 (boiled) was sealed in a glass tube with 11 c.c. of hydrogen; at the end of six hours, at a temperature of 250° , all the hydrogen was absorbed, with the production of water and sulphurous acid. The reaction takes place also at the ordinary temperature, but it is much slower; it does not occur with dilute acid, and light has no appreciable effect.

Influence of Oxygen upon the Decomposition of the Hydrides by Metals, and especially by Mercury.—M. Berthelot.—Mercury is generally considered to have no particular action on gases with which it may be in contact, with the exception of a few such as chlorine, nitrous acid, &c.; but there are gases which have a slow action, among which are HCl, under certain conditions of temperature and time and in the presence of free oxygen.

Direct Reaction of Sulphuric Acid with Mercury at the Ordinary Temperatures.—M. Berthelot.—Boiled sulphuric acid has a slow action on mercury, forming a sulphate, and giving off sulphurous acid when carbonic acid is passed through it. The reactions described in this paper only take place when the sulphuric acid is at the maximum of concentration.

Influence of Surfusion upon the Congelation-point of the Solutions of Potassium Chloride and Sugar.—F. Raoult.—Previous experiments have shown that the value of K in the equation $C = C'(1 - KS)$ for aqueous solutions of chloride of sodium and alcohol is not constant, as was generally admitted, but that it varies sensibly with the concentration. Further experiments show that this is not the case with aqueous solutions of chloride of potassium and of cane-sugar. The new experiments were carried out with the same apparatus as the former ones and the table of figures shows that the molecular lowering

of the congelation-point varies in a very different manner with regard to the concentration of the substances mentioned.

Action of Water upon Phosphorous Trichloride and Phosphorous Oxychloride.—A. Besson.—The reaction between trichloride of phosphorus and water in excess gives phosphorous acid and hydrochloric acid; but when the trichloride is in excess, several peculiarities are noticed. No matter how we arrange for the reaction between a small quantity of water and PCl_3 , we always find a small quantity of a phosphorous oxychloride resulting from the incomplete reaction $\text{PCl}_3 + \text{H}_2\text{O} = 2\text{HCl} + \text{POCl}_3$. This latter is a solid body of the consistence of paraffin, with a smell similar to that of phosphoric oxychloride or chloride of phosphoryl, POCl_3 . This body is insoluble in the usual solvents, and is analogous in the phosphoric series to NOCl , AsOCl , &c.

Production of Strontium Sulphide by means of Sulphuretted Hydrogen and Strontia or Carbonate of Strontium. Influence of Temperature.—J. R. Mourello.—It was found that temperature had a curious influence on the results in the carrying out of this process. A porcelain tube containing either strontia or carbonate of strontia is placed in a furnace, and sulphuretted hydrogen gas is passed over it, at first in the cold. The tube is then gradually warmed to a bright red heat, taking care that the current of gas is strong enough to carry off the water formed by the reaction. When the transformation is complete, a slow current of dry hydrogen is substituted for the sulphuretted hydrogen; after which the tube is allowed to cool, and the contents, monosulphide of strontium, may be withdrawn; this sulphide is not phosphorescent. At a red heat the formula of the reaction is $\text{SrH}_2 + \text{SrO} = \text{SrS} + \text{H}_2\text{O}$. If the temperature is not sufficiently high, the water condenses in the tube and attacks the sulphide already formed, and a mass has been found containing 22 per cent of strontic hydrate and smelling strongly of sulphuretted hydrogen.

Production of Volatile Fatty Acids from the Washing Waters of Wools.—A. and P. Buisine.—The authors think that the production of these volatile acids is now of commercial importance, and they give a *résumé* of the method of procedure by which they are distilled, entangled with and condensed by watery vapour. The waters are first allowed to ferment for eight days, then boiled to drive off the ammonia, then acidulated with sulphuric acid and distilled. The volatile fatty acids found in the largest quantities are acetic and propionic, but we also get a fair percentage of butyric, valerianic, caproic, and benzoic acids, with traces of formic and caprylic acids and phenol. Amongst other applications, this mixture of raw fatty acids is particularly suitable for the production of acetone, methyl-ethylacetone, and the higher acetones which are comprised by the mixture known as "oil of acetone."

Decomposition of Chloroform, Bromoform, and Chloral by a Solution of Potash.—A. Desgrez.—Bright sunlight has an accelerating, and darkness a retarding, influence on the decomposition of chloroform by this means, but potash without the mediation of water has no effect. Chloral has the same action, but it occurs more rapidly.

NOTES AND QUERIES.

* Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Oxidation of Organic Matter.—I have endeavoured to find if any records exist of observations on the oxidation, by atmospheric oxygen, of organic matter in commercial use, *e.g.*, of rope, wood, gums, caoutchouc, &c. The conditions which favour such action appear to be very different from those in the cases of metals. If any correspondent can give any information they will oblige.—H. V.

Destruction of Moths by Formic Aldehyd.—Formic aldehyd is so powerful a disinfectant, and so destructive to most low forms of life, that I think it will help us to solve the great "moth" problem of the household. I propose to pack up furs and winter coats as soon as they are done with in the spring in a large tin box containing vapour of formic aldehyd; but before doing so I should like some of your correspondents who may be better acquainted with the properties of formic aldehyd than I am to tell me—(1) Will it have any injurious action on the fur? (2) Will it prevent the eggs hatching? (3) Will it kill the maggots when hatched? (4) Will it kill the moths or prevent them from laying eggs? I am sure many readers of the CHEMICAL NEWS will find replies to these questions very useful.—MOTH-EATEN.

MEETINGS FOR THE WEEK.

MONDAY, 3rd.—Society of Chemical Industry, 8. "Standard Methods of Tanning Analysis as adopted by the International Association of Leather Trades Chemists, with remarks thereon," by Prof. H. R. Procter and Dr. J. G. Parker. "Extraction of Tanning Materials at various Temperatures," by Dr. J. G. Parker. "Neatsfoot Oil," by J. H. Coste, F.I.C., and E. J. Parry, B.Sc., F.I.C.

TUESDAY, January 4th. } Royal Institution, 3. (Christmas Lectures)
THURSDAY, January 6th. } "Principles of the Electric
SATURDAY, January 8th. } Telegraph," by Prof. Oliver Lodge.

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Head of Chemical Department—WILLIAM A. BONE, D.Sc., Ph.D.

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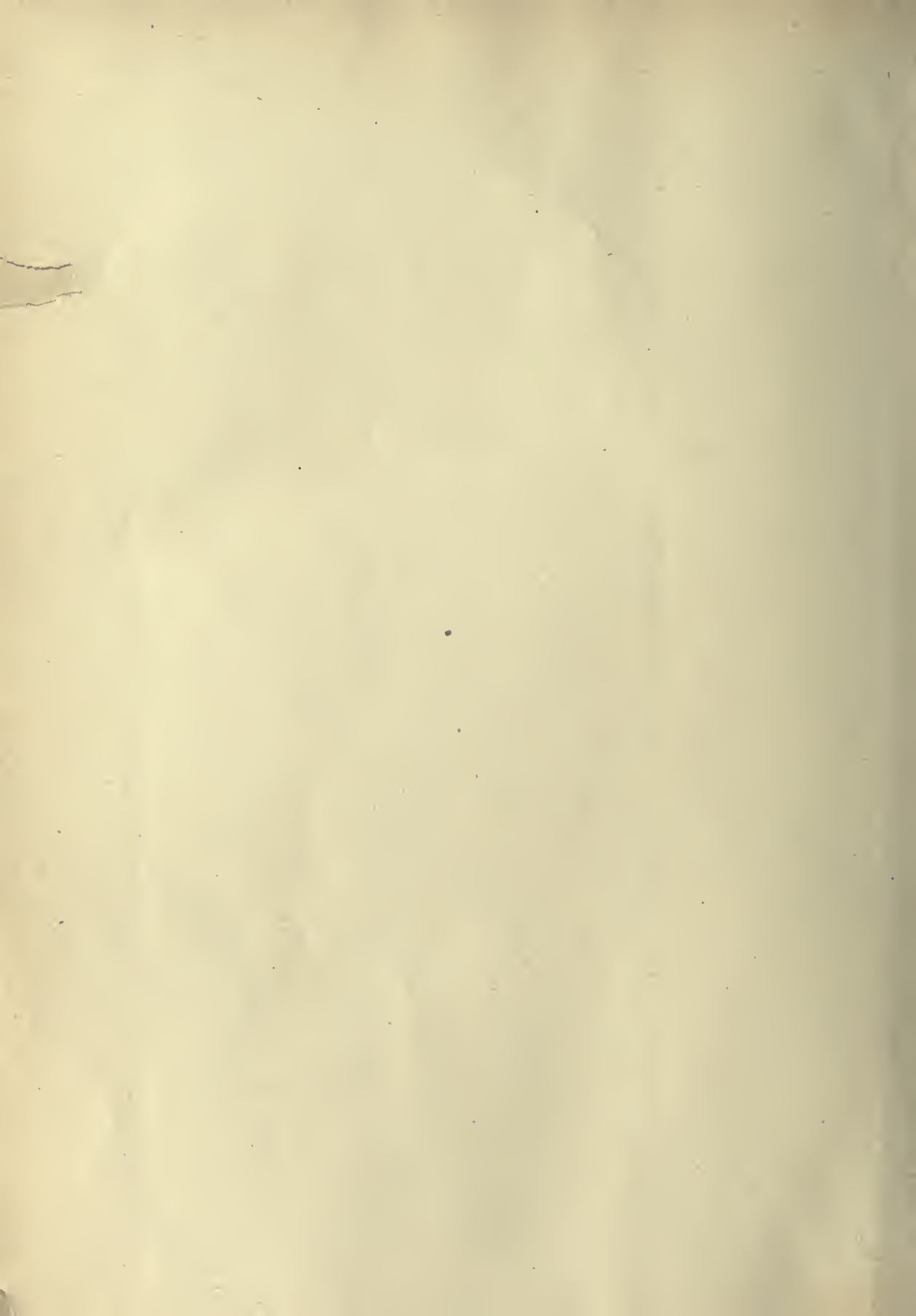
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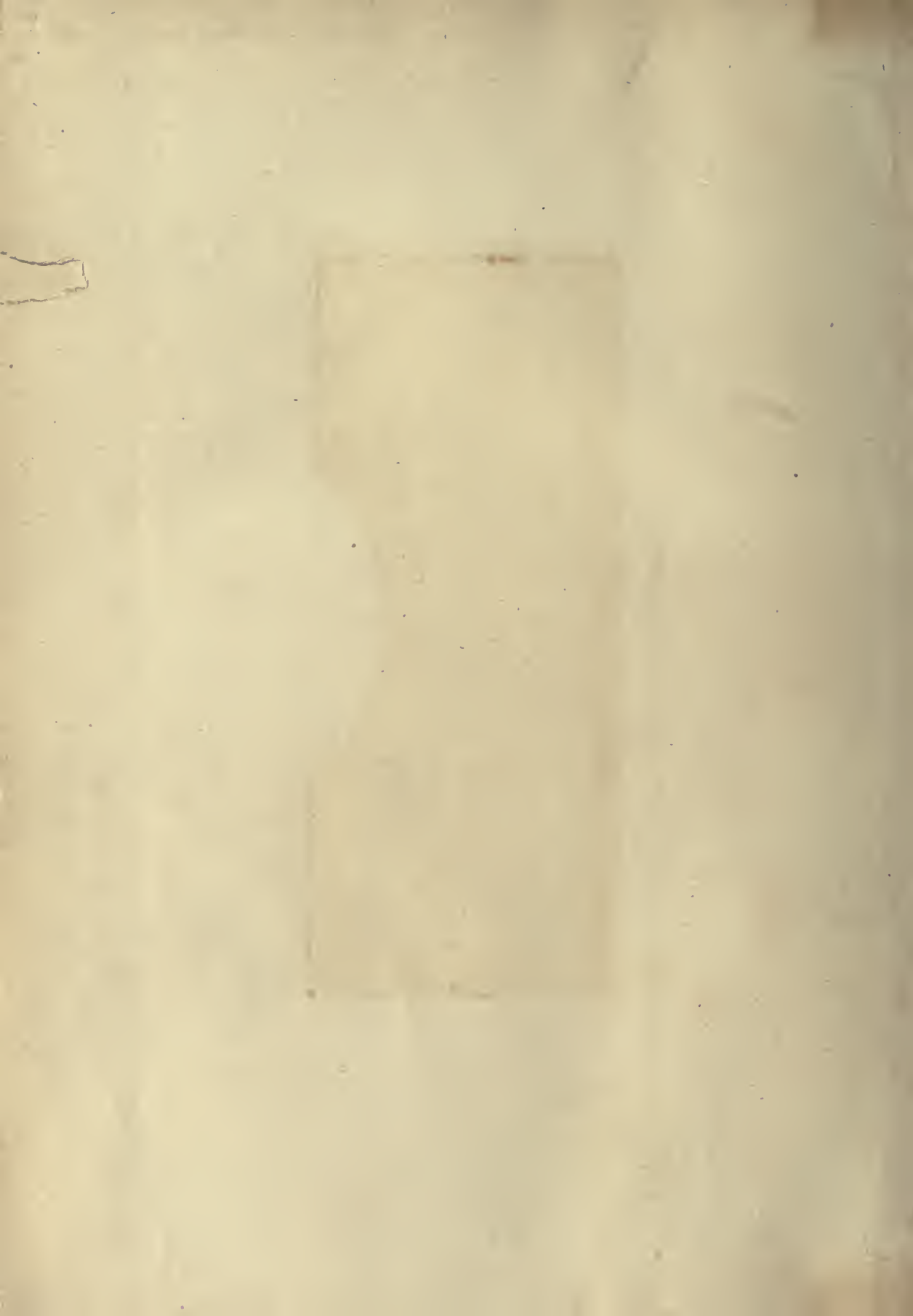
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